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FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* includes those accepted for publication in the ASTM BULLETIN or as Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1950 is given on the last page of this volume. This supplements the list appearing in the 1948 and 1949 *Proceedings* covering all special publications published by the Society up to 1949.

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An Evaluation of Radiography With Particular Emphasis on Detection Methods—C. H. Hastings (see ASTM BULLETIN, *No. 172, February, 1951, p. 66 (TP80)).*

Rotary Screen for Laboratory Separation of Shives from Mechanically Prepared Wood Pulp—H. W. Greider, R. A. MacArthur, and L. C. Gischig (see ASTM BULLETIN, *No. 169, October, 1950, p. 27 (TP213)).*

**Durability Tests of Metalite Sandwich Construction—David G. Reid (see* ASTM BULLETIN, *No. 164, February, 1950, p. 28 (TP40)).*

**Testing of Sandwich Constructions at the Forest Products Laboratory—Edward W. Kuenzi (see* ASTM BULLETIN, *No. 164, February, 1950, p. 21 (TP33)).*

**Preliminary Considerations for Testing Sandwich Radome Materials—G. R. Huisman and R. H. Wight (see* ASTM BULLETIN, *No. 164, February, 1950, p. 19 (TP 31)).*

The Need for Standardization of Industrial Diamond Products—H. L. Strauss, Jr. (see ASTM BULLETIN, *No. 165, April, 1950, p. 57 (TP83)).*

A Reduction-of-Area Gage for Use at Low Temperatures—G. W. Geil and N. L. Carwile (see ASTM BULLETIN, *No. 163, January, 1950, p. 75 (TP25)).*

The Design and Interpretation of Interlaboratory Test Programs—Grant Wernimont (see ASTM BULLETIN, *No. 166, May, 1950, p. 45 (TP107)).*

Comparative Tests in a Single Laboratory—W. J. Youden (see ASTM BULLETIN, *No. 166, May, 1950, p. 48 (TP110)).*

Symposium on High Additive Content Oils

*Summary of Proceedings of the Symposium on High Additive Content Oils (Special Technical Publication No. 102) 1428

Symposium on Turbine Oils

*Summary of Proceedings of the Symposium on Turbine Oils (Special Technical Publication No. 105) 1429

Symposium on Identification and Classification of Soils

Summary of Proceedings of the Symposium on Identification and Classification of Soils (Special Technical Publication No. 113.) 1430

Symposium on Triaxial Testing

*Summary of Proceedings of the Symposium on Triaxial Testing of Soils and Bituminous Mixtures (Special Technical Publication No. 106) 1431

Symposium on Application of Statistics

*Summary of Proceedings of the Symposium on Application of Statistics (Special Technical Publication No. 103) 1432

Symposium on Dynamic Stress Determinations

*Summary of Proceedings of the Symposium on Dynamic Stress Determinations (Special Technical Publication No. 104) 1433

Symposium on the Rôle of Non-Destructive Testing in the Economics of Production

Summary of Proceedings of the Symposium on the Rôle of Non-Destructive Testing in the Economics of Production (Special Technical Publication No. 112). 1434

Symposium on Ultrasonic Testing

Summary of Proceedings of the Symposium on Ultrasonic Testing (Special Technical Publication No. 101) 1435

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List of Special Technical Publications 1490

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* These papers were presented at the First Pacific Area National Meeting of the Society San Francisco, Calif., October 10–14, 1949.

SUMMARY OF PROCEEDINGS

OF THE

FIFTY-THIRD ANNUAL MEETING

Atlantic City, N. J., June 26–30, 1950

This summary of the Fifty-third Annual Meeting of the American Society for Testing Materials, held at the Chalfonte-Haddon Hall Hotel, Atlantic City, N. J., June 26–30, 1950, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 21 technical sessions and 2 informal sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1160; committee members, 637; guests, 334; total 2131; ladies, 408.

The Proceedings are set forth session by session. There were 62 reports and 88 formal papers presented. The record with respect to each has been briefed, the recommendations in the reports having been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported as "adopted as standard" or "adopted as standard, revisions in" it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society. The various recommendations so recorded are included in the Society letter ballot.[1] The actions designated as "accepted as tentative" or "accepted as tentative, revisions in," are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the action thereon is recorded:

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership was canvassed on September 30, 1950.

LOCATION OF ACTIONS ON COMMITTEE REPORTS

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- A-3 on Cast Iron 22
- A-5 on Corrosion of Iron and Steel 22
- A-7 on Malleable-Iron Castings 5
- A-9 on Ferro-Alloys 11
- A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys 22

- B-1 on Wire for Electrical Conductors 10
- B-2 on Non-Ferrous Metals and Alloys 9
- B-3 on Corrosion of Non-Ferrous Metals and Alloys 23
- B-4 on Electrical-Heating, Resistance and Related Alloys 9
- B-5 on Copper and Copper Alloys, Cast and Wrought 22
- B-6 on Die-Cast Metals and Alloys 10
- B-7 on Light Metals and Alloys, Cast and Wrought 10
- B-8 on Electrodeposited Metallic Coatings 10
- B-9 on Metal Powders and Metal Powder Products 9

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- C-2 on Magnesium Oxychloride Cements 13
- C-4 on Clay Pipe 13
- C-7 on Lime 21
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- C-11 on Gypsum 21
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- C-16 on Thermal Insulating Materials 21
- C-17 on Asbestos-Cement Products 21
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- D-2 on Petroleum Products and Lubricants 23
- D-3 on Gaseous Fuels 24
- D-4 on Road and Paving Materials 24
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- D-6 on Paper and Paper Products 20
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- D-20 on Plastics 19

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FIRST SESSION—OPENING SESSION; SYMPOSIUM ON CORROSION OF MATERIALS AT ELEVATED TEMPERATURES

MONDAY, JUNE 26, 2 P.M.

SESSION CHAIRMAN: N. L. MOCHEL

FORMAL OPENING OF THE FIFTY-THIRD ANNUAL MEETING BY PRESIDENT J. G. MORROW

President Morrow, in opening the Fifty-third Annual Meeting, discussed very briefly the current activities of the Society. He cited the care with which committees are formed to include personnel representing all interests so each committee is truly representative of the industries involved. He mentioned the extensive preparation of standards and the constant efforts to revise them when necessary to keep abreast of scientific advances.

Mr. Morrow emphasized the great diversity in the Society's work, the international, as well as national, reputation, and the close cooperation now existing with the Armed Services in Washington. He discussed the major strides of the technical committees, summarized briefly the highlights of a tremendously productive year, and reported on the first Pacific Area National Meeting of the Society.

Symposium on Corrosion of Materials at Elevated Temperatures:[2]

Coal Ash Corrosion of Metals at Elevated Temperatures—C. T. Evans, Jr., presented by the author.

Stress Corrosion Tests on Turbo-Supercharger Materials in the Products of Combustion of Leaded Gasoline—G. B. Wilkes, Jr., presented by the author.

Creep as a Surface Dependent Phenomenon—M. R. Pickus and E. R. Parker, presented from manuscript by Mr. Pickus.

The Effect of Environment on the Stress-Rupture Properties of Metals at Elevated Temperatures—O. Cutler Shepard and Willis Schalliol, presented by Mr. Shepard.

Preliminary Studies of the Effect of Oxidizing Sulfurous Atmospheres on the Rupture Strengths of Inconel "X" and Inconel—A. M. Talbot and E. N. Skinner, presented from manuscript by Mr. Talbot.

Hydrogenizing Effect of Steam on Ferrous Alloys at Elevated Temperatures—C. A. Zapffe and F. K. Landgraf, presented from manuscript by Mr. Landgraf.

Oil Ash Corrosion of Metals at Elevated Temperatures—C. T. Evans, Jr., presented from manuscript by the author.

The Growing Rôle of Protective Coatings for Metals in High-Temperature Service—W. N. Harrison, presented by the author.

SECOND SESSION—METHODS OF TESTING, QUALITY CONTROL, APPEARANCE, METALLOGRAPHY

MONDAY, JUNE 26, 8 P.M.

SESSION CHAIRMAN: G. R. GOHN

Committee E-11 on Quality Control of Materials:

Report presented, in the absence of the chairman, by P. J. Smith and the following actions taken:

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[2] Issued as a separate publication *STP No. 108*.

Adopted as Standard:

Rec. Practice for Designating Significant Places in Specified Limiting Values (E 29 - 49 T), with an editorial change substituting "conformance" for "compliance" throughout the text.

Accepted for Publication:

A.S.T.M. Manual on Quality Control of Materials, Sections 1, 2, and 3.

Committee E-12 on Appearance:

Report presented from manuscript by M. Rea Paul, chairman.

Committee E-1 on Methods of Testing:

Report presented, in the absence of the chairman, by P. J. Smith, secretary, and the following actions taken:

Accepted as Tentative:

Method of Verification and Classification of Extensometers

A.S.T.M. Butadiene Boiling Point Range Thermometer to be published as tentative in the Standard Specifications for A.S.T.M. Thermometers (E 1 - 49)

Accepted as Tentative, Revisions in:

Methods of Verification of Testing Machines (E 4 - 47 T)

Methods of Verification of Calibration Devices for Verifying Testing Machines (E 74 - 47 T)

Adopted as Standard, Revisions in:

Spec. for A.S.T.M. Thermometers (E 1 - 49), comprising revision of thermometers 1 F and 26 C, also changes in the text.

The proposed revision of the Standard Method of Test for Brinell Hardness of Metallic Materials (E 10 - 27) in the form of a new tentative method is to be further revised by the committee and will be submitted to the Society through the Administrative Committee on Standards subsequent to the Annual Meeting.

Appendix:

The Mullen Bursting Strength Test as a Means of Determining the Strength of Annealed Aluminum Foil—W. C. Aber and F. M. Howell, presented by Mr. Howell.

Papers:

Study of Deformation at High Strain Rates Using High-Speed Motion Pictures—Herbert I. Fusfeld and Josephine Carr Feder, presented by Mr. Fusfeld.[3]

Effect of Variations in Notch Acuity on the Behavior of Steel in the Charpy Notched-Bar Test—N. A. Kahn, E. A. Imbembo, and F. Ginsberg, presented from manuscript by Mr. Imbembo.

The Influence of Specimen Dimension and Shape on the Results in Tension Impact Testing—D. S. Clark and D. S. Wood, presented by Mr. Clark.

The Influence of Strain Rate on Some Tensile Properties of Steel—D. S. Clark and P. E. Duwez, presented by Mr. Clark.

Direct Explosion Test to Evaluate Load-Carrying Capacity of Welded Steel Structures—G. S. Mikhalapov, presented from manuscript by the author.[4]

Tensile Properties of Some Aircraft Structural Materials at Various Rates of Loading—Richard F. Klinger, presented by Mr. D. A. Shinn.

A Method for Making Tension Tests of Metals Using a Minature Specimen—R. L. Templin and W. C. Aber, presented by Mr. Aber.

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[3] Published in ASTM BULLETIN, No. 170, December, 1950, p. 75 (TP301).

[4] Published in ASTM BULLETIN, No. 172, February 1951, p. 53 (TP67).

THIRD SESSION—SYMPOSIUM ON THE NATURE, OCCURRENCE, AND EFFECTS OF SIGMA PHASE[5]

MONDAY, JUNE 26, 8 P.M.
SESSION CHAIRMAN: E. L. ROBINSON

Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Cr-Ni Steels—E. J. Dulis and G. V. Smith, presented by Mr. Dulis.

Sigma Phase in Several Cast Austenitic Steels—V. T. Malcolm and S. Low, presented by Mr. Low.

X-ray Study of the Sigma Phase in Various Alloy Systems—Pol Duwez and Spencer R. Baen, presented by Mr. Duwez.

Sigma Phase in Chromium-Molybdenum Alloys with Iron or Nickel—John W. Putman, N. J. Grant, and D. S. Bloom, presented from manuscript by Mr. Putman.

The Tetragonality of the Sigma Phase in the Iron-Chromium System—L. Menezes, J. K. Roros, and T. A. Read, presented from manuscript by Mr. Roros.

(*Symposium continued in Sixth Session*)

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[5] Issued as a separate publication *STP No. 110.*

FOURTH SESSION—SYMPOSIUM ON THE RÔLE OF NON-DESTRUCTIVE TESTING IN THE ECONOMICS OF PRODUCTION

TUESDAY, JUNE 27, 9 A.M.

SESSION CO-CHAIRMEN: H. H. LESTER AND L. H. WINKLER

Committee A-7 on Malleable-Iron Castings:

Report presented by W. A. Kennedy, chairman, and the following action taken:

Adopted as Standard:

Spec. for Pearlitic Malleable Iron Castings (A 220 - 48 T), as revised.

It was announced that the committee had withdrawn from the report, as preprinted, the recommendation for adoption as standard of the Tentative Specifications for Malleable Iron Flanges, Pipe Fittings, and Valve Parts (A 277 - 44 T).

Committee E-7 on Non-Destructive Testing:

Report presented by J. H. Bly, chairman, and accepted as a report of progress.

Symposium on the Rôle of Non-Destructive Testing in the Economics of Production:[6]

A Basic Guide for Management's Choice of Non-Destructive Tests—Robert C. McMaster and Samuel A. Wenk, presented from manuscript by Mr. McMaster.

Defects or Discontinuities That Can Be Revealed in Cast and Wrought Metal Products by Non-Destructive Tests—Kent R. Van Horn, presented informally by the author.

The Rôle of Non-Destructive Testing in the Economics of Castings—J. W. Juppenlatz, presented from manuscript by the author.

(Symposium continued in Seventh Session)

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[6] Issued as a separate publication *STP No. 112.*

FIFTH SESSION—LUNCHEON, PRESIDENT'S ADDRESS, REPORT OF BOARD OF DIRECTORS, AWARD OF HONORARY MEMBERSHIPS, AND INTRODUCTION OF NEW OFFICERS

TUESDAY, JUNE 27, 12:15 P.M.

CHAIRMAN: PAST-PRESIDENT R. L. TEMPLIN

The precedent established at the Annual Meeting a year ago was continued with the scheduling of a luncheon as an official session, at which over 200 members and guests were present.

Presidential Address:

The chairman presented the President, J. G. Morrow, who delivered a brief presidential address.

Report of Board of Directors:

The Executive Secretary made a brief presentation of the Annual Report of the Board of Directors, which had been preprinted. On motion duly seconded the report was accepted for publication in the *Proceedings*.

Award of Honorary Memberships:

The Executive Secretary read citations for the following six members who had been elected by the Board of Directors to Honorary Membership in the Society:

P. H. Bates
W. C. Hanna
Dean Harvey
Prevost Hubbard
Robert Job
H. S. Rawdon

Messrs. Hubbard, Job, and Rawdon were unable to be present at the meeting; the remaining three, Messrs. Bates, Hanna, and Harvey, were at the speaker's table and were individually presented to President Morrow to receive at his hands the certificate of Honorary Membership. Each responded briefly, expressing appreciation of the honor that had been given to him.

Election of Officers:

Results of the letter ballot on election of new officers were announced by the

Chairman of the Tellers' Committee, Henry Grinsfelder. Of the 1179 ballots cast, the results were as follows:

For President, to serve for one year:
  L. J. Markwardt, 1179 votes.
For Vice-President, to serve for two years:
  T. S. Fuller, 1179 votes.
For Directors, to serve for three years:
  B. A. Anderton, 1178 votes.
  R. H. Brown, 1177 votes
  D. K. Crampton, 1178 votes
  H. G. Miller, 1178 votes
  J. R. Trimble, 1177 votes

The newly-elected members of the Board of Directors who were present were introduced, as were the Vice-President-elect and President-elect, who each spoke briefly.

The chariman presented the Past-President's pin to President Morrow for his use after he retires as President at the close of the Annual Meeting.

SIXTH SESSION—SYMPOSIUM ON THE NATURE, OCCURRENCE, AND EFFECTS OF SIGMA PHASE

(Continued from Third Session)

TUESDAY, JUNE 27, 2:30 P.M.
SESSION CHAIRMAN: F. B. FOLEY

Committee E-4 on Metallography:

Report presented by L. L. Wyman, chairman, and accepted as a report of progress. Mr. Wyman introduced G. E. Pelissier who presented the first progress report of Subcommittee XI on Electron Microstructure of Steel. This report with the "Summary of Techniques Employed in the Electron Microscope Study of Steel" was accepted for publication.

Jt. Committee on Effect of Temperature on the Properties of Metals:

Report presented from manuscript by E. L. Robinson, chairman.

Symposium on the Nature, Occurrence, and Effects of Sigma Phase:[5]

This session was a continuation of the Symposium on Effect of Sigma Phase on the High-Temperature Properties of Metals. The papers presented at this session are listed below:

The Formation of Sigma Phase in 17 per cent Chromium Steel—J. J. Heger, presented by the author.

Sigma Phase and Other Effects of Prolonged Heating at Elevated Temperatures on 25 per cent Chromium-20 per cent Nickel Steel—G. N. Emmanuel, presented by the author.

The Occurrence of the Sigma Phase and Its Effect on Certain Properties of Cast Fe-Ni-Cr Alloys—J. H. Jackson, presented from manuscript by the author.

The Formation of Sigma and Its Influence on the Behavior of Stabilized 18 per cent Chromium, 8 per cent Nickel Steels in Concentrated Nitric Acid—R. S. Stewart and Stephen F. Urban, presented from manuscript by Mr. Stewart.

Some Notes on the Structure and Impact Resistance of Columbium-Bearing 18-8 Steels After Exposure to Elevated Temperatures—W. O. Binder, presented by the author.

Observations of the Effect of Sigma on the Mechanical Properties of Columbium Stabilized Weldments in Austenitic Stainless Steels—F. W. Schmitz and M. A. Scheil, presented from manuscript by Mr. Schmitz.

SEVENTH SESSION—SYMPOSIUM ON THE RÔLE OF NON-DESTRUCTIVE TESTING IN THE ECONOMICS OF PRODUCTION[6]

(Continued from Fourth Session)

TUESDAY, JUNE 27, 2:30 P.M.
SESSION CO-CHAIRMEN: R. C. MCMASTER AND J. R. TRIMBLE

This session was a continuation of the Symposium on the Rôle of Non-Destructive Testing in the Economics of Production.

The papers presented at this session are:

The Economics and Practical Application of Cobalt 60 in the Radiographic Inspection of Steel Weldments—W. L. Schwinn, presented from manuscript by the author.

The Economics of Wrought Steel Inspection—C. D. Moriarty, presented from manuscript by the author.

Management's Responsibility for Insistence on Non-Destructive Testing in the Development of New Engineering Products and Processes—L. W. Ball, presented from manuscript by the author.

EIGHTH SESSION—MARBURG LECTURE; DUDLEY MEDAL AWARD; RICHARD TEMPLIN AWARD; SAM TOUR AWARD

TUESDAY, JUNE 27, 8 P.M.
SESSION CHAIRMAN: PRESIDENT J. G. MORROW

President Morrow presented Dr. Wallace R. Brode, Associate Director, National Bureau of Standards, Washington, D. C., as the Twenty-fourth Marburg Lecturer, who then delivered the lecture on "Chemical Spectroscopy." Dr. Brode discussed the development and use of spectroscopic methods in analytical control. The use of both emission spectra and absorption spectra procedures were outlined and the method limitations indicated. In colorimetry and color description, the application of these methods were considered as well as the use of spectrophotometer procedures in the infrared and ultraviolet regions of the spectrum. Part of the lecture was devoted to the field of emission spectroscopy, including the development of rapid analysis procedures and the recent advance in photoelectric recording instruments.

President Morrow, in expressing appreciation to Dr. Brode for his very timely and instructive lecture on a subject of particular interest to the A.S.T.M., presented to him, on behalf of the Society, the Edgar Marburg Lecture Certificate.

Medalists and Award Winners:

The Charles B. Dudley Medal, named after the Society's first president and awarded to the author or authors of a paper of outstanding merit constituting an original contribution on research and engineering materials, was presented to B. J. Lazan, Professor and Head, Department of Materials Engineering, Syracuse University, Syracuse, N. Y., for his paper on "Dynamic Creep and Rupture Properties of Temperature-Resistant Materials Under Tensile Fatigue Stress," presented at the 1949 Annual Meeting and published in the 1949 *Proceedings*.

The Richard L. Templin Award, to stimulate research in the development of testing methods and apparatus and to encourage the presentation to the Society of papers describing new and useful testing procedures and apparatus, was given to D. S. Clark and D. S. Wood, Associate Professor of Mechanical Engineering and Research Assistant, respectively, California Institute of Technology, Pasadena, Calif., for their paper on "The Time Delay for the Initiation of Plastic Deformation at Rapidly Applied Constant Stress," presented at the 1949 Annual Meeting and published in the 1949 *Proceedings*. Dr. Clark accepted the award for his associate and himself.

The second Sam Tour Award, to encourage research on the improvement and evaluation of corrosion testing methods and to stimulate the preparation of technical papers in this field, was given to O. B. Ellis, Senior Research Engineer, Research Laboratories, Armco Steel Corp., Middletown, Ohio, for his paper on "Effect of Weather on the Initial Corrosion Rate of Sheet Zinc," presented at the 1949 Annual Meeting and published in the 1949 *Proceedings*.

NINTH SESSION—EFFECT OF TEMPERATURE

WEDNESDAY, JUNE 28, 9:30 A.M.

SESSION CHAIRMAN: H. C. CROSS

Papers:

The Effect of Various Treatments on the Fatigue Strength of Notched S-816 and Timken 16-25-6 at Elevated Temperatures, W. E. Jones and G. B. Wilkes, Jr., presented by Mr. Jones.

Hardened Alloy Steel for Service up to 700 F.—G. V. Smith, W. B. Seens, and E. J. Dulis, presented by Mr. Smith.

The Selection of a Limited Number from Many Possible Conditioning Treatments for Alloys to Achieve Best Coverage and Statistical Evaluation—J. M. Cameron and W. J. Youden, presented from manuscript by Mr. Cameron.

Effect of Rate of Strain on the Flow Stress of Gas Turbine Alloys at 1200 and 1500 F.—M. J. Manjoine, presented from manuscript by the author.

Chromium-Base Alloys—W. L. Havekotte, C. T. Greenidge, and H. C. Cross, presented from manuscript by Mr. Havekotte.

Hardening of Austenitic Stainless Steels by Mechanical Working at Sub-Zero Temperatures—N. A. Zeigler and P. H. Brace, presented by Mr. Brace.

Effect of Temperatures on the Mechanical Properties, Characteristics and Processing of Austenitic Stainless Steels—V. N. Krivobok and A. M. Talbot, presented from manuscript by Mr. Krivobok.

TENTH AND ELEVENTH SESSIONS—SYMPOSIUM ON METHODS OF TESTING SOILS UNDER TRIAXIAL LOADING[7]

Tenth Session

WEDNESDAY, JUNE 28, 9:30 A.M.

SESSION CHAIRMAN: W. G. HOLTZ

The Symposium on Methods of Testing Soils Under Triaxial Loading consisted of the following ten papers as presented in the Tenth and Eleventh Sessions:

A Triaxial Apparatus for Testing Large Soil Specimens—Earl B. Hall, presented by the author.

Shear Testing at the Waterways Experiment Stations—S. J. Johnson, presented from manuscript by the author.

A Triaxial Shear Investigation on a Partially Saturated Soil—Donald W. Taylor, presented from manuscript by the author.

Shear Characteristics of Remolded Earth Materials—A. A. Wagner, presented from manuscript by the author.

The Importance of Natural Controlling Conditions Upon Triaxial Compression Test Conditions—Donald M. Burmister, presented from manuscript by the author.

Eleventh Session

WEDNESDAY, JUNE 28, 2 P.M.

SESSION CHAIRMAN: W. S. HOUSEL

Triaxial Design Correlated with Flexible Pavement Performance in Kansas—H. E. Worley, presented from manuscript by Mr. W. S. Housel.

Application of Triaxial Compression Test Results to Highway Soil Problems—E. S. Barber and C. L. Sawyer, presented from manuscript by Mr. Barber.

Triaxial Testing, Adapted to Soils, Flexible Base, and Foundations—R. J. Hank, presented from manuscript by Mr. A. W. Johnson.

Some Concepts Concerning Triaxial Compression Testing of Asphaltic Paving Mixtures and Subgrade Materials—H. E. Davis and F. N. Hveem, presented from manuscript by Mr. Davis.

Application of Triaxial Testing to the Design of Bituminous Pavements—Norman W. McLeod, presented from manuscript by the author.

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[7] To be issued as a separate publication.

TWELFTH SESSION—FATIGUE TESTING

WEDNESDAY, JUNE 28, 2 P.M.

SESSION CHAIRMAN: R. E. PETERSON

Committee E-9 on Fatigue:

Report presented, in the absence of the chairman, by O. J. Horger and accepted as a report of progress.

Committee B-4 on Electrical Heating, Resistance, and Related Alloys:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Adopted as Standard:

Method of Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring Back Method) (B 155 - 45 T)
Method of Test for Density of Fine Wire and Ribbon for Electronic Devices (B 180 - 45 T)
Method of Test for Strength of Welded Joints of Lead Wires for Electronic Devices and Lamps (B 203 - 45 T)
Method of Test for Surface Flaws in Tungsten Seal Rod and Wire (B 204 - 45 T)
Method of Test for Equivalent Yield Stress of Thermostat Metals (B 191 - 44 T)
Method of Test for Effect of Controlled Atmosphere Upon Alloys in Electric Furnaces (B 181 - 43 T)
Method of Test for Modulus of Elasticity of Thermostat Metals (Cantilever Beam Method) (B 223 - 48 T)
Method of Testing Wire for Supports Used in Electronic Devices and Lamps (B 157 - 45 T)
Method of Testing Fine Round and Flat Wire for Electronic Devices (B 219 - 47 T)
Method of Measuring Residual Stress in Cylindrical Metal-to-Glass Seals (B 218 - 47 T)
Spec. for Round Nickel Wire for Lamps and Electronic Devices (B 175 - 45 T)
Spec. for Chromium-Nickel-Iron Alloy Castings (25-12 Class) for High-Temperature Service (B 190 - 45 T)
Spec. for Nickel-Chromium-Iron Alloy Castings (35-15 Class) for High-Temperature Service (B 207 - 46 T)

Accepted for Publication as Information:

Proposed Method of Test for Relative Thermionic Emissive Properties of Materials Used in Electron Tubes

Papers:

Effects of Grinding and Other Finishing Processes on the Fatigue Strength of Hardened Steel—L. P. Tarasov and H. J. Grover, presented by Mr. Tarasov.
Effect of Chromium Plating on the Endurance Limit of Steels Used in Aircraft—Hugh L. Logan, presented from manuscript by the author.
Effects of Anodic Coatings on the Fatigue Strength of Aluminum Alloys—G. W. Stickley and F. M. Howell, presented by Mr. Stickley.
Combined Tension-Torsion Creep-Time Relations for Aluminum Alloy 2S-O—Joseph Marin, J. H. Faupel, and L. W. Hu, presented by Mr. Marin.
The Reverting of Hard-Drawn Copper to Soft Condition Under Variable Stress—John N. Kenyon, presented from manuscript by the author.

THIRTEENTH SESSION—NON-FERROUS METALS, SPECTROSCOPY

THURSDAY, JUNE 29, 9 A.M.

SESSION CHAIRMAN: L. L. WYMAN

Committee E-2 on Emission Spectroscopy:

Report presented from manuscript by J. H. Coulliette and accepted as a report of progress.

Committee B-9 on Metal Powder and Metal Powder Products:

Report presented by W. A. Reich, chairman, and the following action taken:

Accepted as Tentative, Revision in:

Spec. for Sintered Metal Powder Structural Parts (B 222 - 47 T)

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by Bruce W. Gonser, chairman, and the following actions taken:

Reaffirmation of Standards:

Spec. for Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 - 42)
Spec. for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 - 43)
Spec. for Nickel (B 39 - 22)
Spec. for Rolled Zinc (B 69 - 39)

Spec. for Lead-Coated Copper Sheet (B 101 – 40)
Spec. for Electrolytic Cathode Copper (B 115 – 43)

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by I. V. Williams, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum-Alloy Die Forgings

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 48 T)
Spec. for Aluminum-Base Alloys for Permanent Mold Castings (B 108 – 49 T)
Spec. for Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 – 49 T)
Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 49 T)
Spec. for Aluminum and Aluminum-Alloy Sheet and Plate (B 209 – 49 T)
Spec. for Aluminum-Alloys Drawn Seamless Tubing (B 210 – 49 T)
Spec. for Aluminum - Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 48 T)
Spec. for Aluminum-Alloy Extruded Tubes (B 235 – 49 T)
Spec. for Aluminum-Alloy Pipe (B 241 – 49 T)

Editorial Changes Accepted in:

Spec. for Magnesium-Base Alloy Sand Castings (B 80 – 49 T)
Spec. for Magnesium-Base Alloy Sheet (B 90 – 49 T)
Spec. for Magnesium-Base Alloy Forgings (B 91 – 49 T)
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 49 T)
Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 49 T)
Spec. for Aluminum and Aluminum-Alloy Bars, Rods and Wire (B 211 – 49 T)
Spec. for Magnesium-Base Alloy Extruded Round Tubing (B 217 – 49 T)
Spec. for Aluminum and Aluminum-Alloy Extruded Bars, Rods and Shapes (B 221 – 49 T)
Spec. for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 48 T)

Committee B-1 on Wires for Electrical Conductors:

Report presented, in the absence of the chairman, by W. R. Hibbard, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (B 172 – 49 T)
Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric Stranded Members for Electrical Conductors (B 173 – 49 T)
Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 49 T)
Spec. for Hard-Drawn Aluminum Wire for Electrical Purposes (B 230 – 49 T)
Spec. for Concentric-Lay-Stranded Aluminum Conductors Steel-Reinforced (B 232 – 48 T)

Adopted as Standard, Revisions in:

Spec. for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (B 8 – 49)
Spec. for Cored, Annular, Concentric-Lay-Stranded Copper Conductors (B 226 – 49)
Spec. for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 – 46)
Spec. for Hot-Rolled Copper Rods for Electrical Purposes (B 49 – 41)
Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 – 48)

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented, in the absence of the chairman, by J. S. Pettibone, and accepted as a report of progress.

Committee B-6 on Die-Cast Metals and Alloys:

Report presented, in the absence of the chairman, by J. W. Caum, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Die Castings (B 85 – 49 T)

Adopted as Standard:

Spec. for Copper-Base (Brass) Alloy Die Castings (B 176 – 49 T)

Papers:

Magnesium-Cerium Wrought Alloys for Elevated-Temperature Service—K. Grube, J. A. Davis, and L. W. Eastwood, presented by Mr. Eastwood.

Magnesium-Cerium Cast Alloys for Elevated-Temperature Service—K. Grube and L. W. Eastwood, presented from manuscript by Mr. Eastwood.

Aluminum-6 per cent Magnesium Alloys for Elevated-Temperature Service—L. W. Eastwood, Webster Hodge, and C. H. Lorig, presented from manuscript by Mr. Eastwood.

FOURTEENTH SESSION—STEEL, FERRO-ALLOYS, CHEMICAL ANALYSIS

THURSDAY, JUNE 29, 9 A.M.

SESSION CHAIRMAN: T. S. FULLER

Committee A-9 on Ferro-Alloys:

Report presented by W. C. Bowden, Jr., chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Molybdenum Salts and Compounds (A 146 - 49 T)
Spec. for Ferroboron (A 323 - 49 T)
Spec. for Ferrotitanium (A 324 - 49 T)

Adopted as Standard, Revisions in:

Spec. for Spiegeleisen (A 98 - 41)
Spec. for Ferromanganese (A 99 - 39)
Spec. for Ferrosilicon (A 100 - 39)
Spec. for Ferrochromium (A 101 - 42)
Spec. for Ferrovanadium (A 102 - 39)
Spec. for Ferromolybdenum (A 132 - 39)
Spec. for Ferrotungsten (A 144 - 38)

In all the above specifications, Committee A-9 submitted an editorial change in the revision, as preprinted, involving a change in footnote (*a*) under the revised table on chemical requirements changing the phrase "For purposes of determining compliance to these specifications, an observed or a calculated value obtained from analysis shall be rounded off to . . ." to read "For purposes of determining compliance to these specifications, the reported analysis shall be rounded off to . . ."

Committee A-1 on Steel:

Report presented by N. L. Mochel, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Steel Sheet Piling
Spec. for Heat-Treated Steel Tires
Methods and Definitions for Mechanical Testing of Steel Bars

Accepted as Tentative, Revisionsin:

Spec. for Hot-Rolled Carbon-Steel Bars (A 107 - 49 T)
Spec. for Cold-Finished Carbon-Steel Bars and Shafting (A 108 - 49 T)
Spec. for Alloy-Steel Bolting for High-Temperature Service (A 193 - 49 T)
Spec. for Carbon and Alloy Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 - 49 T)
Spec. for Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292 - 49 T)
Spec. for Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 - 49 T)
Spec. for Carbon and Alloy Steel Forgings for Turbine Bucket Wheels (A 294 - 49 T)
Spec. for Steel Machine Bolts and Nuts and Tap Bolts (A 307 - 49 T)
Spec. for Structural Steel for Ships (A 131 - 49 T)

The committee withdrew from the report as preprinted the recommendation that the following tentatives be adopted as standard and recommends that they be revised as indicated and continued as tentative:

Spec. for General Requirements for Delivery of Rolled Steel Plates, Shapes, and Bars for Structural Use (A 6 - 49 T)
Spec. for Steel for Bridges and Buildings (A 7 - 49 T)
Spec. for Structural Silicon Steel (A 94 - 49 T), with a correction in Section 6, changing Paragraph (*b*) to (*c*) in the report, as preprinted.
Spec. for Structural Steel for Locomotives and Cars (A 113 - 49a T)
Spec. for Structural Rivet Steel (A 141 - 49 T)
Spec. for High-Strength Structural Rivet Steel (A 195 - 49a T)
Spec. for Low-Alloy Structural Steel (A 242 - 49 T)
Spec. for Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality

(Plates 2 in. and Under in Thickness) (A 283 - 49 T)
Spec. for Low and Intermediate Tensile Strength Carbon-Silicon Steel Plates for Machine Parts and General Construction (A 284 - 49 T)

Adopted as Standard, Revision in:

Spec. for Factory-Made Carbon-Steel and Carbon-Molybdenum-Steel Welded Fittings (A 234 - 44)
Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 - 47), jointly with Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys.

Editorial Changes Accepted in:

Spec. for Welded and Seamless Steel Pipe (A 53 - 47)
Spec. for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 - 46), with a correction in Table I, consisting of changing the word "maximum" to "minimum" copper content of 0.20 per cent.
Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 - 46)
Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 - 46)
Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 - 47)
Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 47)
Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 47)
Spec. for Welded Alloy-Steel Boiler and Superheater Tubes (A 249 - 47)
Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 - 47)
Spec. for Seamless Cold-Drawn Low-Carbon Steel Heat Exchanger and Condenser Tubes (A 179 - 46)
Spec. for Seamless Cold-Drawn Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 - 46)

Committee E-3 on Chemical Analysis of Metals:

Report presented by D. R. Evans, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Rec. Practices for Apparatus and Reagents for Chemical Analysis of Metals (E 50 - 48) with the following changes in the report, as preprinted:

Section 6.—In the fourth sentence change the word "protective" to "suitable gas," and change "available for" to "worn when". At the end of the sixth sentence add "before removing the shipping caps." Change the beginning of the seventh sentence to read "Adequate properly ventilated storage space . . ."

Change Paragraph (*g*) to read "(*g*) *Metallic Mercury* gives off an appreciable amount of vapor and must not be handled in quantity in a confined space even at room temperature. Every effort should be made to avoid spilling mercury since it easily lodges in cracks and crevices and may result in mercury poisoning."

In the second sentence in Item (*2*) of Paragraph (*h*), delete "cleaning down the condensed acid and for" and insert "and interior of the hood" after the word "flue." In the first sentence of Item (*3*) on *Destruction of Organic Matter* delete the words "The hood of." Also in Item (*3*) delete "cause loss of time and material by deflagration or explosion" in the second sentence and replace by "deflagrate or explode."

Table I.—Under "Percentage of Reagent by Weight" in the "Min." and "Max." columns, add ".0" to the values for hydrobromic, hydrochloric, hydrofluoric, nitric, perchloric, and sulfuric acids.

Section 8.—In the description of Reagent No. 2 under "*Preparation*" change the second sentence to read "Cool and dilute with HNO_3 (1:9) to 1 liter in a volumetric flask."

Delete the Section on Reagent No. 3 since there is no change from the present method.

A new paragraph should be added for Reagent No. 6 in which there is a footnote error. Change "p. 220 (1936)" to "p. 1065 (1926)."

Concerning Reagent No. 24, delete the first sentence in Method A and replace by "Transfer 0.1000 g. of manganese metal to a 150 ml. beaker and add 10 ml. of HNO_3 (1:1). Heat gently to dissolve the sample and expel the brown fumes."

Under new Reagent No. 113, add "and filter." at the end of the sentence.

Change the section on Reagent No. 121 to read "Method A (*Preparation from Barium Diphenylamine Sulfate*).—Dissolve 0.32 g. of barium diphenylamine sulfonate in 100 ml. of hot water. Add 0.5 g. of Na_2SO_4, stir, and filter off the precipitate of $BaSO_4$. Store in a dark colored bottle."

"Method B (*Preparation from Sodium Diphenylamine Sulfonate*).—Dissolve 0.20 g. of sodium diphenylamine sulfonate in 100 ml. of water. Store in a dark colored bottle."

Footnotes:—Editorial changes in footnotes in Method E 50, as preprinted, involve the inclusion of title of papers in footnotes 7, 7[c], 7[d], and 7[e].

Withdrawal of Standard and Tentatives:

Photometric Method for Determination of Bismuth in Pig Lead (E 58 - 45 T)
Photometric Method for Determination of Iron in 70-30 Copper-Nickel Alloy (E 63 - 46 T)
Method of Sampling Slab Zinc (Spelter) (E 65 - 48)

Reference is made in the report to a number of additional recommendations on standards to be presented to the Society through the Administrative Committee on Standards. All of the new and revised methods are to be issued in a new 1950 edition of the Book of A.S.T.M. Methods of Chemical Analysis of Metals.

Papers:

Reproducibility of Results of Tension Tests on Specimens Prepared from Cast Steel Coupons—Harry A. Schwartz and W. Kenneth Bock, presented by Mr. Schwartz.

Some Effects of Austenitic Grain Size and Metallurgical Structure on the Mechanical Properties of Steel—G. M. Sinclair and T. J. Dolan, presented by Mr. Sinclair.

Rockwell C Hardness on Cylindrical Steel Specimens—Frank W. Hussey, presented by the author.

Welding Characteristics of Open Hearth and Bessemer Seamless Pipe—A. B. Wilder, W. B. Kennedy, and F. W. Crouch, presented from manuscript by Mr. Wilder.

Welds Between Dissimilar Alloys in Full-Size Steam Piping—R. U. Blaser, F. Eberle, and J. T. Tucker, Jr., presented from manuscript by Mr. Eberle.

Some Considerations in the Joining of Dissimilar Metals for High-Temperature High-Pressure Service—O. R. Carpenter, N. C. Jessen, J. L. Oberg, and R. D. Wylie, presented from manuscript by Mr. Carpenter.

FIFTEENTH SESSION—CEMENT AND CONCRETE, ROOFING MATERIALS

THURSDAY, JUNE 29, 9 A.M.
SESSION CHAIRMAN: H. F. GONNERMAN

Committee C-1 on Cement:

Report presented by F. H. Jackson, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Determining the Bleeding of Cement Pastes and Mortars, with Section 7 (*b*) revised to read as follows:

"(*b*). *Mortars.*—Immediately following the flow test, the mortar remaining in the bowl shall be mixed for 30 sec. with one hand, protected with a rubber glove, after which the glove shall be freed of adhering mortar. Within a total elapsed time of not more than 2 min. and 30 sec. after completion of the original mixing of the mortar batch, placing of the mortar in the sample container shall be started. The placing shall be done in three layers, each layer tamped 40 times with the tamper and the surface struck off level with the edge of the 5 in. diameter container by screeding with a steel straight edge. The remainder of the test shall be conducted in the same manner as that for the paste, Section (*a*), using as a starting time the completion of striking off the surface of the mortar in the container".[8]

Adopted as Standard, Revisions in:

Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 - 49)
Spec. for Portland Cement (C 150 - 49), revision added to the report as preprinted:[8]

Table II.—In footnote "*c*," delete all except the first two sentences and add the following: "Unless otherwise specified, the strength tests for types I and II cements will be made only at 3 and 7 days and for type III cement only at 1 and 3 days. The strength at any age designated herein shall be higher than the strength at any preceding age."

Committee C-2 on Magnesium Oxychloride Cements:

Report presented from manuscript by H. F. Gonnerman, session chairman.

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by K. B. Woods, chairman, and accepted as a report of progress.

Committee C-4 on Clay Pipe:

Report presented, in the absence of the chairman, by H. F. Gonnerman and the following action taken:

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[8] This recommendation, not preprinted and subject to unanimous affirmative letter ballot of Committee C-1 was not accepted. However, subsequent to the Annual Meeting, the revision was accepted by the Administrative Committee on Standards as a tentative revision on September 26, 1950.

Adopted as Standard:

Spec. for Standard Strength Perforated Clay Pipe (C 211 - 47 T)

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented, in the absence of the chairman, by E. H. Berger, vice-chairman, and the following actions taken:

Accepted as Tentative:

Definitions of Terms Relating to Bituminous Waterproofing and Roofing Materials

Accepted as Tentative, Revisions in:

Spec. for Asphalt Roofing Surfaced with Powdered Talc or Mica (D 224 - 46 T), with a correction in the report as preprinted, substituting the word "*mineral*" for "*material*" in Section 2.

Spec. for Asphalt Shingles with Mineral Granules (D 225 - 46)

Spec. for Asphalt Roofing Surfaced with Mineral Granules (D 249 - 46 T), with a correction in the report as preprinted, replacing the word "*material*" with "*mineral*" in Section 2.

Spec. for Wide Selvage Asphalt Roofing Surfaced with Mineral Granules (D 371 - 46)

Spec. for Asphalt Siding Surfaced with Mineral Granules (D 699 - 46)

Reapproval of Standards:[9]

Spec. for Woven Cotton Fabrics Saturated with Bituminous Substances (D 173 - 44)

Spec. for Asphalt for Use in Built-Up Roof Coverings (D 312 - 44)

Papers:

Exploratory Tests to Develop a Method for Determining the Air Content of Hardened Concrete—Alexander Klein, David Pirtz, and Milos Polivka, presented from manuscript by Mr. H. E. Davis.

Pulse Techniques Applied to Dynamic Testing—J. R. Leslie, presented by the author.

Method of Petrographic Examination of Aggregates for Concrete—Katherine Mather and Bryant Mather, presented by Mr. Mather.

Results on Testing Surface Waterproofers—F. O. Anderegg, presented by the author.[10]

Testing Cement Base Paints and Dampproofers—William M. Spurgeon, presented from manuscript by the author.[11]

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[9] Added to the report.

[10] ASTM BULLETIN No. 171, January, 1951, p. 48(TP10).

[11] *Ibid.*, p. 53 (TP15).

SIXTEENTH SESSION—LUNCHEON, PRESENTATION OF AWARDS OF MERIT, AND RECOGNITION OF FORTY- AND FIFTY-YEAR MEMBERS

THURSDAY, JUNE 29, 12:15 P.M.

CHAIRMAN: PRESIDENT J. G. MORROW

This luncheon session was attended by over 150 members and guests. At the conclusion of the luncheon the chairman introduced Past-President T. A. Boyd, Chairman of the Committee on Award of Merit. Mr. Boyd stated that he and his associates on the Award of Merit Committee, namely, Messrs. D. E. Parsons, C. H. Rose, H. S. Vassar, and L. H. Winkler, had the honor and distinction of making the selections and nominations to the Board of Directors for the first recipients of the recently-established A.S.T.M. Award of Merit. After explaining the procedures that had been followed, beginning with consideration of recommendations from the technical committees of the Society, he then introduced to the President, with appropriate citation for each, the following recipients of the award:

Alfred P. S. Bellis
Harold F. Dodge
A. W. Dow
H. P. Ferguson
D. W. Kessler
T. G. Stitt
T. Smith Taylor
Percy H. Walker
LeRoy L. Wyman

All but Mr. Walker were present and received personally the certificate of award.

Presentation of Forty- and Fifty-Year Members:

The Executive Secretary presented brief citations for six 50-year members, five of whom were present or represented and to whom were tendered by the

President specially engrossed framed certificates.

The Executive Secretary also cited the 25 members who this year have reached 40 years of membership. Nine were present and received the appropriate forty-year certificate.

The President introduced a guest and member of the Society from Brazil, Mr. Mariano J. M. Ferraz, Director General of the Sorocabana Railroad Materials Co. and President of the Federation of Industries of Sao Paulo. Mr. Ferraz extended greetings to the Society from the Brazilian Standards Association and invited close cooperation between the two societies in their mutual interest in advancement of standardization activities.

The chairman introduced Past-President Arthur W. Carpenter, referring to the fact that this meeting brings to a close eight consecutive years of service by Mr. Carpenter on the Board of Directors. Mr. Carpenter responded most appropriately to this introduction, expressing his appreciation at the opportunity to have served the Society as he has been privileged to do.

SEVENTEENTH SESSION—BUILDING MATERIALS

THURSDAY, JUNE 29, 2:30 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT L. J. MARKWARDT

Committee C-22 on Porcelain Enamel:

Report presented from manuscript by W. N. Harrison, chairman.

Committee C-21 on Ceramic Whitewares:

Report presented, in the absence of the chairman, by J. W. Caum and the following action taken:

Accepted as Tentative:

Definitions of Terms Relating to Ceramic Whitewares

Committee C-19 on Structural Sandwich Constructions:

Report presented from manuscript by A. G. H. Dietz, chairman.

Committee C-14 on Glass and Glass Products:

Report presented from manuscript by E. W. Tillotson, chairman, and the following actions taken:

Accepted as Standard, Revisions in:

Method of Hydrostatic Pressure Test on Glass Containers (C 147 – 43)
Method of Polariscopic Examination of Glass Containers (C 148 – 43)
Method of Thermal Shock Test on Glass Containers (C 149 – 43)

Committee C-18 on Natural Building Stone:

Report presented by L. W. Currier, chairman, and the following actions taken:

Adopted as Standard:

Definitions of Terms Relating to Natural Building Stone (C 119 – 48 T)

Adopted as Standard, Revisions in:

Method of Test for Compressive Strength of Natural Building Stone (C 170 – 46)

Withdrawal of Standards:

Method of Test for Modulus of Elasticity of Natural Building Stone (C 100 – 36)
Method of Shear Testing of Natural Building Stone (C 102 – 36)

Committee C-15 on Manufactured Masonry Units:

Report presented by J. W. Whittemore, chairman, and the following actions taken:

Adopted as Standard:

Spec. for Facing Brick (C 216 – 48 T)

Adopted as Standard, Revisions in:

Spec. for Building Brick (Made from Clay or Shale) (C 62 – 49)
Spec. for Sewer Brick (Made from Clay or Shale) (C 32 – 42)
Spec. for Structural Clay Load-Bearing Wall Tile (C 34 – 49)
Spec. for Structural Clay Non-Load-Bearing Wall Tile (C 56 – 49)
Spec. for Structural Clay Floor Tile (C 57 – 49)
Methods of Sampling and Testing Brick (C 67 – 44)
Definitions of Terms Relating to Structural Clay Tile (C 43 – 49)

Committee D-7 on Wood:

Report presented by L. J. Markwardt, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service
Method of Test for Ash in Wood
Methods of Test for Water Solubility of Wood
Method of Test for Ether Solubility of Wood
Method of Test for Ether Solubility of Wood
Method of Test for Alcohol-Benzene Solubility of Wood
Method of Test for One Per Cent Caustic Soda Solubility of Wood
Method of Test for Holocellulose in Wood
Method of Test for Lignin in Wood
Method of Test for Alpha-Cellulose in Cellulosic Materials
Method for Preparation of Extractive-Free Wood

Adopted as Standard:

Spec. for Chromated Zinc Chloride (D 1032 – 49 T)
Spec. for Tanalith (D 1034 – 49 T)
Spec. for Creosote-Coal-Tar Solution (D 391 – 49 T)
Spec. for Zinc Chloride (D 432 – 49 T)
Methods of Chemical Analysis of Chromated Zinc Chloride (D 1033 – 49 T)
Methods of Chemical Analysis of Tanalith (D 1035 – 49 T)
Methods of Chemical Analysis of Zinc Chloride (D 199 – 49 T)

Adopted as Standard, Revision in:

Methods of Testing Small Clear Specimens of Timber (D 143 – 49)

EIGHTEENTH SESSION—PAINT, ELECTRICAL INSULATING MATERIALS, COAL AND COKE, SOAP, TEXTILES

THURSDAY, JUNE 29, 2:30 P.M.

SESSION CHAIRMAN: R. D. BONNEY

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Pure Para Red Toner (D 475 – — T)
Method of Test for Heptane Number of Hydrocarbon Solvents
Method of Test for Kauri-Butanol Value of Hydrocarbon Solvents
Method of Test for Nitrocellulose Diluting Power of Hydrocarbon Solvents
Methods for Chemical Analysis of Blue Pigments
Methods for Chemical Analysis of Yellow, Orange, and Green Pigments Containing Lead Chromate, and Chromium Oxide Green (D 126 – — T)
Methods for Chemical Analysis of Zinc Dust (Metallic Zinc Powder) (D 521 – — T)
Methods of Testing Varnishes (D 154 – — T)

Accepted as Tentative, Revisions in:

Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 – 46 T)
Spec. for Venetian Red (D 767 – 48 T)
Methods of Testing Drying Oils (D 555 – 47), comprising new tests for hydroxyl value and break.
Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 47), comprising new definitions of the terms spar varnish, sealer, primer and size.

Adopted as Standard:

Spec. for Methods of Testing Ethylcellulose (D 914 – 47 T)
Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 48 T)

Adopted as Standard, Revisions in:

Methods of Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese (D 50 – 36)
Methods of Testing Varnishes (D 154 – 47)
Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 47), new definition of oil color.
Spec. and Test for Soluble Nitrocellulose (D 301 – 48)

Withdrawal of Standards:

Methods of Chemical Analysis of Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments, and Chrome Green (D 126 – 36)
Methods of Chemical Analysis of Zinc Dust (D 521 – 40)

Editorial Changes Accepted in:

Spec. for Red and Brown Iron Oxide Pigments (D 84 – 48 T)
Method of Test for Night Visibility of Traffic Paints (D 1011 – 49 T)

Committee D-16 on Industrial Aromatic Hydrocarbons:

Report presented, in the absence of the chairman, by R. B. Greene, secretary, and the following actions taken:

Adopted as Standard:

Method of Test for Thiophene in Benzene (D 931 - 47 T)

Adopted as Standard, Revisions in:

Spec. for Nitration Grade Benzene (D 835 - 47)
Spec. for Industrial Grade Benzene (D 836 - 47)
Spec. for Industrial 90 Benzene (D 837 - 47)
Spec. for Refined Solvent Naphtha (D 838 - 47)
Spec. for Crude Light Solvent Naphtha (D 839 - 47)
Spec. for Crude Heavy Solvent Naphtha (D 840 - 47)
Spec. for Nitration Grade Toluene (D 841 - 47)
Spec. for Industrial Grade Toluene (D 842 - 47)
Spec. for Nitration Grade Xylene (D 843 - 47)
Spec. for Industrial Grade Xylene (D 844 - 47)
Spec. for Five-Degree Xylene (D 845 - 47)
Spec. for Ten-Degree Xylene (D 846 - 47)
Method of Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 - 47), with a further change to retain the present thermometers as alternate.

Editorial Change Accepted in:

Method of Test for Solidifying Point of Benzene (D 852 - 47)

Committee D-9 on Electrical Insulating Materials

Report presented, in the absence of the chairman, by O. E. Anderson, recording secretary, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Orange Shellac and Other Indian Lacs for Electrical Insulation (D 784 - 44 T)
Spec. for Black Bias-Cut Varnished Cloth Tape Used for Electrical Insulation (D 373 - 49 T)
Methods of Testing Nonrigid Polyvinyl Tubing (D 876 - 46 T)
Methods of Testing Electrical Insulating Oils (D 117 - 47 T), with the addition of Sections 26 and 27 on Definitions and Significance of Power Factor.
Methods of Testing Askarels (D 901 - 49 T)
Methods of Testing Glass-Bonded Mica for Electrical Insulation (D 1039 - 48 T)
Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 49 T)
Methods of Testing Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 - 47 T)
Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 49 T), with two editorial changes: in Section 15, add the word "about" in the phrase "a whole unit of about the same area"; in Section 56, change item 2 to read "Average retention of tearing strength or folding endurance after baking, expressed as a percentage of the results obtained on the unbaked specimens, and".
Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 49)

Adopted as Standard, Revisions in:

Methods of Testing Shellac Used for Electrical Insulation (D 411 - 44)

It was announced that the committee had withdrawn from the report, as preprinted, the proposed new Tentative Specifications for Cellulose Acetate Sheet and Film for Electrical Insulation, jointly with Committee D-20 on Plastics.

Committee D-5 on Coal and Coke:

Report presented, in the absence of the chairman, by C. H. Sawyer, secretary, and the following actions taken:

Adopted as Standard, Revisions in:

Method of Test for Sieve Analysis of Coke (D 293 - 29), with new footnote 3.
Method of Tumbler Test for Coke (D 294 - 29), with new footnote 2^a:

> "Users' attention is called to the fact that yields of certain sizes as determined by use of specified screens cannot be compared directly with results obtained by using screens as specified prior to the 1950 revision. An important example occurs in the case of the tumbler test stability factor (per cent remaining on 1 in. after tumbling) which will be slightly higher than comparable previous results using the formerly specified 1.06 in. screen."

Committee D-12 on Soaps and Other Detergents:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary, and the following actions taken:

Accepted as Tentative:

Spec. for Solid Soap for Low-Temperature Washing (Low and Medium Titer)
Spec. for Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer)

Adopted as Standard:

Spec. for Borax (D 929 - 47 T)

Adopted as Standard, Revision in:

Definition of Terms Relating to Soaps and Other Detergents (D 459 - 48), with the deletion of "synthetics" and addition of "synthetic detergents" at the end of the definition of "alkaline detergent."

Editorial Changes Accepted in:

Spec. for Alkaline Soap Powder (D 534 - 42)

Accepted for Publication as Information:

Proposed Method of Test for pH of Aqueous Solutions of Soaps and Detergents
Proposed Method of Test for Foaming Properties of Surface Active Agents

Committee D-13 on Textile Materials:

Report presented by H. J. Ball, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Vegetable Matter in Scoured Wool
Spec. for Mechanical Sheet Felt
Method of Test for Resistance of Pile Floor Coverings to Insect Pest Damage
Method of Test for Snag Resistance of Hosiery
Methods of Testing Bonded Fabrics
Method of Test for Magnetic Rating of Asbestos Used for Electrical Purposes

Accepted as Tentative, Revisions in:

Definitions of Terms Relating to Textile Materials (D 123 - 49 T), comprising new definitions of the terms: bleaching; binding; carding; extensibility; deformation, delayed or creep; creep, primary; creep, secondary; yarn, Kraft; hair; and noil. The Committee withdrew from the report the definitions for: water repellency (textile); water resistance (fabric); narrow fabrics and take-up.
Method of Test for Fastness of Colored Textiles to Light (D 506 - 45 T)
Method of Test for Resistance of Textile Fabrics to Water (D 583 - 40 T)
Methods of Test for Resistance of Textile Materials to Microorganisms (D 684 - 45 T)
Methods of Test for Fineness of Wool (D 419 - 47 T)
Spec. and Methods of Test for Fineness of Wool Tops (D 472 - 47 T)
Method of Test for Determination of Small Amounts of Copper, Manganese and Nickel in Textiles (D 377 - 47 T)
Spec. and Methods for Asbestos Yarns (D 299 - 49 T)
Spec. and Methods for Asbestos Lap (D 1061 - 49 T)
Methods of Testing and Tolerances for Continuous Filament Rayon and Estron Yarns (D 258 - 48 T)
Methods of Testing and Tolerances for Glass Yarn (D 578 - 49 T)
Spec. and Methods for Asbestos Roving for Electrical Purposes (D 375 - 49)
Methods of Testing Rayon and Estron Staple (D 540 - 44)
Methods of Testing Felt (D 461 - 49)

Adopted as Standard:

Spec. for Woven Asbestos Cloth (D 677 - 46 T), as revised
Definition of Terms Relating to Textile Materials (D 123 - 49 T), comprising revisions in 11 definitions and a revised Table I on Synthetic fibers and monofilaments. The proposed revision of the definition of the term "yarn, single" was withdrawn by the committee.

Adopted as Standard, Revisions in:

Definition of Terms Relating to Textile Materials (D 123 - 49)
Recommended Practice for a Universal System of Yarn Numbering (D 861 - 47)
Method of Test for Hard Scoured Wool in Wool in the Grease (D 584 - 47)
Spec. for Mechanical Roll Felt (D 944 - 49)
Methods of Testing Felt (D 461 - 49)

Withdrawal of Tentative:

Spec. for Bleached Wide Cotton Sheeting (D 503 - 48 T)

Editorial Changes Accepted in:

Methods of Testing and Tolerances for Cotton Yarns (D 180 - 49 T)
Methods of Testing and Tolerances for Continuous Filament Rayon and Estron Yarns (D 258 - 48 T)
Methods of Testing and Tolerances for Rayon Tire Cord (D 885 - 46 T)

The recommendation that the Tentative Methods of Test for Resistance of Textile Materials to Microorganisms (D 684 - 45 T) be revised was withdrawn from the report; also the recommendation that the Tentative Methods of Test for Evaluating Treated Textiles for Permanence of Resistance to Microorganisms (D 862 - 45 T) be discontinued was withdrawn from the report.

At the conclusion of the report, the session took cognizance of the retirement of Professor Ball as chairman of Committee D-13 after 20 years of service.

NINETEENTH SESSION—SYMPOSIUM ON IDENTIFICATION AND CLASSIFICATION OF SOILS

THURSDAY, JUNE 29, 8 P.M.

SESSION CHAIRMAN: G. W. MCALPIN

Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following action taken:

Adopted as Standard, Revision in:

Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 – 45)

Symposium on Identification and Classification of Soils:[2]

Identification and Classification of Soils—An Appraisal and Statement of Principles—Donald M. Burmister, presented from manuscript by the author.

A Standard Classification of Soils as Used by the Bureau of Reclamation—E. A. Abdun-Nur, presented from manuscript by the author.

Discussion of the Classification of Highway Subgrade Materials Initiated by the Highway Research Board—W. F. Abercrombie, presented from manuscript by the author.

Soil Classification for Highway Purposes—Edward A. Willis, presented from manuscript by the author.

Soil Series Names as Identified by the Division of Soil Survey as a Basis for Interpretive Soil Classifications for Engineering Purposes —Earl J. Felt, presented from manuscript by the author.

Pilot Studies of Soil Density Measurements by Means of X-rays—D. Berdan and R. K. Bernhard, presented by Mr. Bernhard.

TWENTIETH SESSION—PLASTICS, ADHESIVES, SHIPPING CONTAINERS

FRIDAY, JUNE 30, 9:30 A.M.

SESSION CHAIRMAN: R. E. HESS

Committee D-14 on Adhesives:

Report presented by Frank W. Reinhart, chairman, and accepted as a report of progress.

Committee D-20 on Plastics:

Report presented, in the absence of the chairman, by B. L. Lewis, secretary, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Injection Molding of Specimens of Thermoplastic Materials (D 1130 - 50 T)

Accepted as Tentative, Revisions in:

Spec. for Melamine-Formaldehyde Molding Compounds (D 704 – 44 T)

Spec. for Molds for Test Specimens of Plastic Molding Materials (D 647 - 49 T)

Adopted as Standard:

Spec. for Vinyl Chloride-Acetate Resin Plastic Sheets (D 708 – 44 T)

Spec. for Vinyl Chloride-Acetate Resin Molding Compounds (D 728 – 44 T)

Spec. for Cast Allyl Plastic Sheet, Rods, Tubes, and Shapes (D 819 – 45 T)

Tentative Method of Test for Luminous Reflectance, Transmittance and Color of Materials. (D 791 - 44 T), with deletions as follows: Delete "which is simpler, more rapid, or less expensive" in Section 1(*a*); delete "mu" in Section 2(*e*). In Section 5 Paragraphs (*b*) and (*c*) should be designated (*a*) and (*b*) under 2. In Note 3, denominator of equation for R_i should be squared.

Method of Test for Measuring the Molding Index of Thermosetting Molding Powder (D 731 – 48 T)

Method of Test for Stiffness in Flexure of Plastic (D 747 – 48 T)

Method of Test for Resistance of Plastics to Accelerated Service Conditions (D 756 – 46 T)

Method of Test for Specific Gravity of Plastics (D 792 – 48 T)

Method of Test for Apparent Density and Bulk Factor of Nonpouring Molding Powders (D 954 – 48 T)

Method of Test for Measuring Shrinkage from Mold Dimensions of Molded Plastics (D 955 – 48 T)

Rec. Practice for Molding Specimens of Amino Plastics (D 956 – 48 T)

Rec. Practice for Determining Mold Surface Temperature of Commercial Molds for Plastics (D 957 – 48 T)

Rec. Practice for Determining Temperatures of Standard A.S.T.M. Molds for Test Specimens of Plastics (D 958 - 48 T)

Adopted as Standard, Revisions in:

Methods of Test for Index of Refraction of Transparent Organic Plastics (D 542 - 42)
Method of Test for Surface Irregularities of Flat Transparent Plastic Sheets (D 637 - 43)

Withdrawal of Tentative:

Spec. for Nonrigid Vinyl Butyral Plastics (D 745 - 44 T)

Committee D-20 withdrew from its report, as preprinted, the new Tentative Specifications for Cellulose Acetate Sheet and Film for Electrical Insulation, jointly with Committee D-9 on Electrical Insulating Materials.

Committee D-20 had recommended the adoption as standard the Tentative Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 48 T), subject to concurrence of Committee D-9 since this method is under the joint jurisdiction of both committees. This recommendation is under consideration in Committee D-9.

The committee also withdrew from its report the recommendation that Tentative Specifications for Methacrylate Molding Compounds (D 788 - 48 T) and Tentative Method of Test for Weight Loss of Plastics on Heating (D 948 -47 T) be adopted as standard.

Committee D-10 on Shipping Containers:

Report presented, in the absence of the chairman, by T. A. Carlson, and the following actions taken:

Adopted as Standard:

Method of Drop Test for Bags (D 959 - 48 T)
Def. of Terms Relating to Shipping Containers (D 996 - 48 T)
Method of Drop Test for Cylindrical Shipping Containers (D 997 - 48 T)

Adopted as Standard, Revision in:

Method of Incline Impact Test for Shipping Containers (D 880 - 47)

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Test for Rosin Oils

Accepted as Tentative, Revisions in:

Method of Test for Water in Liquid Naval Stores (D 890 - 46 T)

Adopted as Standard:

Method of Test for Volatile Oil in Rosin (D 889 - 46 T), as revised with a further change in Section 3(*b*) to delete the last sentence of this paragraph.

It was announced that the committee had withdrawn from the report, as preprinted, the recommendations for revision and adoption as standard of the Tentative Methods of Test for Acid Number of Rosin (D 465 - 49 T) and the Tentative Method of Test for Saponification Number of Rosin (D 464 - 49 T).

Committee D-6 on Paper and Paper Products:

Report presented by W. R. Willets, chairman, and the following actions taken:

Accepted as Tentative:

Method of Static Bending Test for Corrugated Paperboard
Test for Water-Soluble Sulfates in Paper and Paperboard
Spec. for Filter Paper for Use in Chemical Analysis

Accepted as Tentative, Revisions in:

Test for Organic Nitrogen in Paper and Paper board (D 982 - 48 T)
Test for Scuff Resistance of Paperboard (D 1029 - 49 T), with editorial changes, substituting "peeling resistance" for "scuff resistance" throughout the text.

Adopted as Standard:

Test for Paraffin Wax Absorptiveness of Paper (D 983 - 48 T)
Test for Reducible Sulfur in Paper (D 984 - 48 T)
Test for 45-deg., 0-deg. Directional Reflectance for Blue Light (Brightness) of Paper (D 985 - 48 T)
Method of Preparation of a Magnesium Oxide Standard for Spectral Reflectivity (D 986 - 48 T)

Adopted as Standard, Revisions in:

Method of Test for Basis Weight of Paper and Paper Products (D 646 - 44)
Method of Test for Hydrogen Ion Concentration (pH) of Paper Extract (D 778 - 46)

Papers:

The Measurement of Dynamic Modulus in Adhesive Joints at Ultrasonic Frequencies—A. G. H. Dietz, P. J. Closmann, G. M. Kavanagh, and J. N. Rossen, presented from manuscript by Mr. Rossen.

Stress Cracking of Polyethylene—R. H. Carey, presented from manuscript by the author.[12]

Effects of Elevated Temperatures on Strength of Thermosetting Plastic Laminates—N. Fried, R. R. Winans, and L. E. Sieffert presented by Mr. Fried.

Outdoor Weather Aging of Plastics Under Various Climatological Conditions—S. E. Yustein, R. R. Winans, and H. J. Stark, presented by Mr. Yustein.[13]

Rotary Screen for Laboratory Separation of Shives from Mechanically Prepared Wood Pulp—H. W. Greider, R. A. MacArthur, and L. C. Gischig, presented by Mr. Greider.[14]

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[12] Published in ASTM BULLETIN, No. 167, July, 1950, p. 56 (TP 138).

[13] Published in ASTM BULLETIN, No. 173, April, 1951, p. 31 (TP 85).

[14] Published in ASTM BULLETIN, No. 169, October, 1950, p. 27 (TP213).

TWENTY-FIRST SESSION—BUILDING MATERIALS, FIRE TESTS OF MATERIALS

FRIDAY, JUNE 30, 2 P.M.
SESSION CHAIRMAN: L. J. TROSTEL

Committee C-11 on Gypsum:

Report presented by L. S. Wells, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Def. of Terms Relating to Gypsum (C 11 - 48)
Spec. for Gypsum (C 22 - 41)
Methods of Testing Gypsum and Gypsum Products (C 26 - 42)
Spec. for Gypsum Plasters (C 28 - 40), with a revision in Section 17 to read: "*Fineness.*—Gypsum gauging plaster for finish coat shall all pass a No. 14 mesh (1410-micron) sieve and not less than 60 per cent shall pass a No. 100 (149-micron) sieve.
Spec. for Gypsum Wallboard (C 36 - 42)
Spec. for Gypsum Lath (C 37 - 42)
Spec. for Gypsum Molding Plaster (C 59 - 40)
Spec. for Keene's Cement (C 61 - 40)
Spec. for Gypsum Sheathing Board (C 79 - 42)

Committee C-7 on Lime:

Report presented from manuscript by W. C. Voss, chairman.

Committee C-17 on Asbestos-Cement Products:

Report presented from manuscript by H. R. Snoke, chairman.

Committee C-8 on Refractories:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Accepted as Tentative, Revision in:

Spec. for Castable Refractories for Boiler Furnaces and Incinerators (C 213 - 47 T)
Spec. for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (C 63 - 47)
Methods of Chemical Analysis of Refractory Materials (C 18 - 45)
Def. of Terms Relating to Refractories (C 71 - 49)

Accepted for Publication as Information:

Proposed Method of Test for Disintegration of Fireclay Refractories in an Atmosphere of Carbon Monoxide

Committee C-16 on Thermal Insulating Materials:

Report presented, in the absence of the chairman, by J. S. Pettibone and the following actions taken:

Adopted as Standard, Revisions in:

Methods of Test for Thickness and Density of Blanket or Batt Type Thermal Insulating Materials (C 167 - 44)
Def. of Terms Relating to Thermal Insulating Materials (C 168 - 48)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented, in the absence of the chairman, by R. C. Corson and the following actions taken:

Accepted as Tentative:

Method of the Fire Hazard Classification of Building Materials

Accepted as Tentative, Revisions in:

Methods of Fire Tests of Building Construction and Materials (E 119 - 47), including the proposed change in Section 26 (*c*) which was retained as a tentative revision.

Adopted as Standard, Revisions in:

Methods of Fire Test of Building Construction and Materials (E 119 - 47), with the exception of Section 26 (*c*) which was accepted as a tentative revision.
Method of Test for Combustible Properties of Treated Wood by the Crib Test Method (E 160 - 46)

Method of Tests for Combustible Properties of Treated Wood by the Fire Tube Test Method (E 69 – 47)

Papers:

The Effect of Time and Temperature of Burning on the Properties of Quicklime Prepared from Calcite—J. A. Murray, H. C. Fischer, and D. W. Sabean, presented by Mr. Murray.

Automatic Control of Thermal Conductivity Apparatus—E. M. Herrmann, R. B. Plate, and W. P. Sinclair, presented from manuscript by Mr. Plate.[15]

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[15] Published in ASTM BULLETIN, No. 170, December, 1950, p. 69 (TP295).

TWENTY-SECOND SESSION—CORROSION OF FERROUS AND NON-FERROUS METALS, CAST IRON, WATER

FRIDAY, JUNE 30, 2 P.M.

SESSION CHAIRMAN: H. L. MAXWELL

Committee D-19 on Industrial Water:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary, and the following actions taken:

Accepted as Tentative:

Methods of Test for Sodium and Potassium in Industrial Water
Methods of Test for Electrical Conductivity of Industrial Water
Methods of Test for Hardness in Industrial Water
Method of Identification of Types of Microorganisms in Industrial Water

Accepted as Tentative, Revisions in:

Methods of Test for Total Carbon Dioxide, and Calculation of the Carbonate and Bicarbonate Ions in Industrial Water (D 513 – 48 T)
Methods of Test for Silica in Industrial Water (D 859 – 47 T)
Rec. Practice for Application of X-Ray Diffraction Method (D 934 – 47 T)

Adopted as Standard:

Def. of Terms Relating to Industrial Water
Method of Test for Total Aluminum Ion in Industrial Water (D 857 – 48 T), as revised

Adopted as Standard, Revisions in:

Method of Reporting Results of Examination and Analysis of Water-Formed Deposits (D 933 – 49)

Jt. Committee on Boiler Feedwater Studies:

Report presented, in the absence of the chairman, by R. E. Hess and accepted for publication.

Committee A-3 on Cast Iron:

Report presented by J. S. Vanick, chairman, and the following actions taken:

Accepted as Tentative:

Methods of Impact Testing of Cast Iron

Reaffirmation of Standards:

Spec. for Cast Iron Culvert Pipe (A 142 – 38)
Spec. for Gray Iron Castings for Valves, Flanges and Pipe Fittings (A 126 – 42)
Spec. for Cast Iron Pit-Cast Pipe for Water or Other Liquids (A 44 – 41)
Spec. for Cast Iron Soil Pipe and Fittings (A 74 – 42)

Committee A-5 on Corrosion of Iron and Steel:

Report presented, in the absence of the chairman, by H. F. Harmon, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (A 93 – 48 T)
Spec. for Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 – 47 T)

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel and Related Alloys:

Report presented, in the absence of the chairman, by F. L. LaQue and the following actions taken:

Adopted as Standard, Revision in:

Spec. for Seamless and Welded Austenitic Steel Sanitary Tubing (A 270 – 47), jointly with Committee A-1 on Steel

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented in the absence of the chairman, by J. W. Caum and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Cartridge Brass Sheet, Strip, Plate, Bar and Disks (B 19 – 49 T)

- Spec. for Naval Brass Rods, Bars, and Shapes (B 21 - 49 T)
- Spec. for Brass Plate, Sheet and Strip (B 36 - 49 T)
- Spec. for Leaded Brass Plate, Sheet and Strip (B 121 - 49 T)
- Spec. for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet and Strip (B 122 - 49 T)
- Spec. for Gilding Metal Strip (B 130 - 49 T)
- Spec. for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 49 T)
- Spec. for Copper-Zinc-Alloy Rods and Bars (B 151 - 49 T)
- Spec. for Copper Sheet, Strip, and Plate (B 152 - 49 T)
- Spec. for Aluminum Bronze Plate, Sheet and Strip (B 169 - 49 T)
- Spec. for Beryllium-Copper Alloy Plate, Sheet, and Strip (B 194 - 49 T)
- Spec. for Beryllium-Copper Alloy Strip, Special Grade (B 195 - 49 T)
- Spec. for Beryllium-Copper Alloy Wire (B 197 - 49 T)
- Spec. for Copper-Nickel Zinc Alloy Wire (B 206 - 49 T)

Adopted as Standard, Revisions in:

- Spec. for Seamless Copper Water Tube (B 88 - 49)
- Spec. for Copper-Silicon Alloy Plate, Sheet and Strip for General Purposes (B 97 - 49)
- Spec. for Phosphor Bronze Plate, Sheet and Strip (B 103 - 49)
- Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 49)
- Spec. for Brass Wire (B 134 - 49)
- Spec. for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 49)
- Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 - 45) with a change in the revision of Section 1, as preprinted, deleting the revision of the first sentence "This method describes the technique for conducting the mercurous nitrate testing of commercial cast copper and wrought copper and copper alloy products intended for industrial use."

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented, in the absence of the chairman, by F. L. LaQue and accepted as a report of progress.

Papers:

- The Forming Characteristics of Beryllium Copper Strip—John T. Richards and Ellsworth M. Smith, presented from manuscript by Mr. Richards.
- Spray Testing with Natural and Synthetic Sea Water. Part I. Corrosion Characteristics in the Testing of Metals—Thomas P. May, and A. L. Alexander, presented from manuscript by Mr. May.
- Spray Testing with Natural and Synthetic Sea Water. Part II. A Study of Organic Coatings —A. L. Alexander and Thomas P. May, presented by Mr. Alexander.
- Impact Deposition of Atmospheric Sea Salts on a Test Plate—Alfred H. Woodcock, presented from manuscript by the author.

TWENTY-THIRD SESSION—PETROLEUM PRODUCTS, FUELS, ROAD MATERIALS, RUBBER

FRIDAY, JUNE 30, 2 P.M.

SESSION CHAIRMAN: A. W. CARPENTER

Committee D-2 on Petroleum Products and Lubricants:

Report presented by O. L. Maag, acting chairman, and the following actions taken:

Accepted as Tentative:

- Method of Test for Apparent Viscosity of Lubricating Grease
- Method of Test for Boiling Point Range of Polymerization-Grade Butadiene
- Method of Test for Carbonyl Content of Butadiene
- Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases, with appropriate revisions of certain of the volume correction factors.
- Methods of Test for Phosphorus in Lubricating Oils, Lubricating Oil Additives, and Their Concentrates
- Method of Test for Analysis of 60 Octane Number *Iso*octane, Normal Heptane A.S.T.M. Knock-Test Reference Fuel Blends by Infrared Spectrophotometry
- Method of Test for 1,3-Butadiene in C_4 Hydrocarbon Mixtures by Ultraviolet Spectrophotometry
- Method of Test for Acidity of Residue from Distillation of Gasoline and of Petroleum Solvents
- Method of Test for Water Tolerance of Aircraft Fuels

Accepted as Tentative, Revisions in:

Spec. for Gasoline (D 439 - 49 T)
Spec. for Diesel Fuel Oils (D 975 - 48 T)
Spec. for Aviation Gasolines (D 910 - 48 T)
Method of Test for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water (D 665 - 49 T)
Method of Test for Sulfur in Petroleum Products by Lamp-Gravimetric Method (D 90 - 47 T)
Method of Test for Chlorine in Lubricating Oils by Bomb Method (D 808 - 49 T)
Method of Chemical Analysis for Phosphorus in Lubricating Oils (D 809 - 44 T)
Method of Test for Determination of Purity from Freezing Points (D 1016 - 49 T)
Method of Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 - 48 T)
Method of Test for Free and Corrosive Sulfur in Petroleum Products (D 130 - 48 T)

Adopted as Standard:

Method of Test for Interfacial Tension of Oil Against Water by the Ring Method (D 971 - 48 T)
Method of Test for Oxygen Stability of Lubricating Greases by the Oxygen Bomb Method (D 942 - 47 T)
Method of Test for Determination of Butadiene Content of Polymerization-Grade Butadiene (D 973 - 48 T)
Method of Test for Saponification Number of Petroleum Products by Potentiometric Titration (D 939 - 47 T), as revised

Adopted as Standard, Revisions in:

Method of Test for Thermal Value of Fuel Oil (D 240 - 39)
Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (D 129 - 49)
Method of Test for Existent Gum in Gasoline (D 381 - 49)

It was announced that the committee had withdrawn from the report as preprinted the proposed adoption as standard of Tentative Method for Test for Foaming Characteristics of Crankcase Oils (D 892 - 45 T).

Withdrawal of Standard:

Method of Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline (D 130 - 30)

Accepted for Publication as Information:

Proposed Test for Hydrocarbon Type Analysis of Diesel Fuels by Silica Gel Adsorption
Proposed Test for Bromine Number of Petroleum Distillates (Color Indicator Method)
Proposed Test for Bromine Number of Petroleum Distillates (Electrometric Method)
Proposed Test for Measurement of Refractive Dispersion of Hydrocarbon Liquids
Proposed Test for Measurement of Density of Knock Test Standard Normal Heptane and *Iso*octane by the Pycnometer
Proposed Test for Pour Stability Characteristics of Winter Grade Motor Oils
Proposed Test for Reduced Pressure Distillation of Petroleum Products
Proposed Definition and Specifications for Tractor Fuels[5]

Committee D-4 on Road and Paving Materials:

Report presented by F. H. Baumann, chairman, and the following actions taken:

Accepted as Tentative:

Method of Test for Bitumen Content of Paving Mixtures by Centrifuge, with a new heading for Section 5 and the procedure for mixtures with tar binders, now Section 6, becoming a note.

Adopted as Standard, Revision in:

Spec. for Asphalt Plank (D 517 - 40)

Committee D-4 recommended the adoption as standard of the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 42 T), subject to concurrence of Committee D-18 on Soils since this method is under joint jurisdiction of both committees. Committee D-18, however, recommended that Method D 698 be continued as tentative.

Committee D-3 on Gaseous Fuels:

Report presented, in the absence of the chairman, by R. E. Hess, Assistant Executive Secretary.

Committee D-15 on Engine Antifreezes:

Report presented by H. R. Wolf, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Hydrometer-Thermometer Field Tester for Engine Antifreezes
Method of Test for Reserve Alkalinity of Concentrated Engine Antifreezes
Method of Test for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer
Method of Test for Water in Concentrated

Engine Antifreezes by the Iodine Reagent Method
Method of Test for Boiling Point of Engine Antifreezes
Method of Test for Ash Content of Concentrated Engine Antifreezes

Paper:

The Determination of Freezing Point of Engine Antifreezes (A Report on the Activities of Subcommittee I of Committee D-15)—R. E. Mallonee and F. L. Howard, presented from manuscript by Mr. Mallonee.[16]

Committee D-11 on Rubber and Rubber-Like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (D 27 - 46 T), changing the revision of Section 6 to read: "Change the value of "1 per under" to "1 per cent under for solid conductors and 2 per cent under for stranded conductors" in the permissible variations specified in this section."
Methods of Testing Rubber Hose (D 380 - 49 T)
Methods of Testing Rubber Insulated Wire and Cable (D 470 - 49 T)
Methods of Testing Hard Rubber Products (D 530 - 49 T)
Spec. for Insulated Wire and Cable: Polyvinyl Insulating Compound (D 734 - 43 T), changing the revision of Section 6 to read: "Change the value of "1 per under" to "1 per cent under for solid conductors and 2 per cent under for stranded conductors" in the permissible variations specified in this section."
Spec. for Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (D 754 - 46a T)
Spec. for Insulated Wire and Cable: Performance Synthetic Rubber Compound (D 755 - 46a T)
Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (D 865 - 48 T)
Methods of Chemical Analysis of Rubber Products (D 297 - 43 T)
Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 - 41)
Methods of Testing Flat Rubber Belting (D 378 - 41)

Adopted as Standard, Revisions in:

Spec. for Insulated Wire and Cable: Performance Rubber Compound (D 353 - 48)
Spec. for Insulated Wire and Cable: Heat-Resisting Rubber Compound (D 469 - 46)
Method of Air Pressure Heat Test of Vulcanized Rubber (D 454 - 48)
Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (D 572 - 48)
Method of Testing Compressed Asbestos Sheet Packing (D 733 - 49)

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[16] Published in ASTM BULLETIN, No. 172, February, 1951, p. 43 (TP57).

ANNUAL ADDRESS BY THE PRESIDENT

JAMES G. MORROW[1]

JUNE 27, 1950

An unwritten rule prevails in the A.S.T.M. that the Presiding Officer at each Annual Meeting shall deliver an address. What is said by me today is in compliance with that custom and will be pretty much in the form of an informal talk.

Last year President Templin ably forecast our work for the near future, and in the two preceding years Presidents Boyd and Carpenter, from somewhat different approaches, touched upon the progress in the first 50 years of the Society's history. It remains for me, therefore, only to comment to you on current activities in general.

Our Society was planned and founded by men of vision, rugged honesty, great ability, and fairmindedness. Their experience was broad and their judgment sound. The A.S.T.M. has faithfully maintained the ideals of its founders throughout the years, and today is outstanding and unique in its field.

In forming our committees, great care is exercised in the selection of members so that all interests will be represented in the personnel composing the committee. Long experience has demonstrated that it is impossible for a standard to win general recognition when its committee is not wholly representative of the industries involved.

In A.S.T.M. committees, the freest possible expression of the views of all members—each of whom has an equal voice in the work brought formally to the Society for group consideration and adoption—is the practice. This method of conducting our business gives each consumer, producer, or independent engineer a fair chance to speak and be heard and to present to the respective committees all angles and phases of the problems under consideration.

Much thought is given by each committee in the preparation of its standards. This provides full assurance to the public that the materials purchased and inspected in accordance with the recommendations of this Society will result in furnishing the best that can be produced at the lowest possible cost, and at the same time afford all safety necessary to the public welfare.

Revisions in our many specifications are made from time to time as increased scientific knowledge and experience dictates. Our Society is furnishing much assistance to, and is in close relationship with, various interests using and producing materials in accordance with our specifications. From a meager beginning of $54 in 1902, our sale of publications has risen to an estimated $300,000 in 1950. This indicates the steady growth in the demand for and the usefulness of our standards. The sum mentioned represents slightly more than one half of our total income.

The soundness and competence of our Society's specifications have created an international as well as a national reputation. No engineering society stands higher, and none commands greater respect. The influence of its work is far reaching because its activities are closely attuned to the needs of those whom it serves—whether they be in engineering, in industry, or in government.

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The great diversity of the Society's work—cutting across every major materials field—makes it almost impractical to even briefly cover it. Only the highlights of major A.S.T.M. accomplishments will be referred to, and we shall not attempt to forecast particular trends except to mention, in passing, the vast field which will be ours as science unfolds the applications and uses of isotopes, and on which committee work is now being undertaken. It might be well to mention also the closer relationship and cooperation that now exists with the Armed Services at Washington.

Many of the Society's technical committees have made major strides in various phases of their work. The organization of new technical committees has continued. More consideration, time, and effort than is generally realized is devoted to the formation of new technical groups. In organizing these new groups it must first be established that both producer and consumer will give good support to the project, if A.S.T.M. undertakes it. Then the logical technical division into which the product falls for development must be decided. These unpublished problems require fairminded judgment and consideration.

Our technical groups in their general order are Ferrous Metals; Non-Ferrous Metals; Cement, Ceramic and Related Materials; Miscellaneous Materials, including Paints, Textiles, Petroleum, Gaseous Fuels, Electrical Insulation, Rubber Products, etc.; and also the group dealing with general methods of testing. These committees and their subcommittees are composed of many of the country's leading technical men who have cooperated, inspired, worked and produced outstanding documents which will benefit industry and governments, particularly on this North American Continent. Many worth-while contributions have been made by our committees to our knowledge of the properties and the tests of materials and also in standardizing specifications.

A great amount of work is carried on by these committees. To bring this clearly before you, at the last meeting of Committee A-1 on Steel, it took 2½ hours for subcommittee chairmen to briefly abstract their reports so that Committee A-1 might be able to present a summary of its 1950 work at this Annual Meeting for your approval. Committee A-1 has developed specifications of wide moment and application. They are the so-called Master Specifications for Structural Steels with their allied products. The influence of these specifications will be far-reaching and is indicative of the enormous amount of work and unselfish splendid effort of the members of this and other committees involved in the development of our specifications and research programs.

I have referred to our Steel Committee particularly, but the same applies to all our committees. Without exception each committee, whether it be on paint, rubber, petroleum, plastics, timber, electrical insulation, or the newer committees on such subjects as nondestructive testing, quality control of materials, or asbestos-cement products, has been doing a tremendous amount of work and has accomplished much.

Our working committees who are responsible for all specifications and development and research work, have a membership, eliminating duplication, of more than 2600. This is over one third of our total membership and demonstrates clearly that A.S.T.M. is an organization of workers from whom much has come and from whom much can be expected.

Those who have been in touch with the international setup realize how difficult is the task of international standardization because of different conditions prevailing in different countries and on several continents, and the lack of a common language. The apparent feeling in some countries is that international specifications would militate against their export trade.

The situation, however, offers some promise, and in view of our already successful efforts along certain lines, I feel that we should continue with this important work.

In October of last year, our Society held its first meeting on the West Coast. It was the opinion of those who attended this first Pacific Area National Meeting in San Francisco that it had much the character of an Annual Meeting. There were close to 1000 in attendance, along with 110 ladies. The Technical Sessions (15 in all) were well attended and the papers were excellent. The traditions of the West were maintained with splendid committees, excellent leaders, and lots of enthusiasm. They planned well and worked hard in providing a strong technical program as well as a fine entertainment program. Western Meetings are a "must" for the future.

The work of our Board of Directors and of the Administrative Committees is set out in detail in the Report of the Board of Directors. This is our "Book of Words" for the year. It has been a tremendously productive year. Our technical activities, publications, and meetings are dealt with in their entirety for your information. Your officers and committees have labored faithfully on all fronts. This Board report indicates that their efforts have not been unsuccessful and splendid cooperation has been shown.

In closing, I should like to express my deep appreciation for the splendid help that has been accorded me on every side during my tenure as President. I particularly want to express appreciation to Mr. Warwick and the Headquarters' Staff who have been most considerate, and who are doing such an excellent job. I wish to reiterate that great honor has come to Canada and to me in making me your President and permitting me to serve this great and influential organization. The strong ties already existing between our countries have been fortified by your generous gesture. My thanks to all of you for your exceeding great kindness.

ANNUAL REPORT OF THE BOARD OF DIRECTORS

The Board of Directors presents to the members this annual report of its stewardship of the Society's affairs in an historic year, 1950,—the midway point of the twentieth century. The year also happens to come midway between two important anniversaries of the Society—that in 1948 marking the fiftieth anniversary of the beginnings of the Society in the American Section of the International Association for Testing Materials, and that in 1952 which will mark the fiftieth anniversary of the incorporation of our Society—its real beginning as the organization known throughout the world today by its initials, A.S.T.M.

It seems appropriate, then, to pause briefly for a glance back over these fifty years: years that have seen almost fabulous advances in scientific knowledge and its applications to industry and down into the daily lives of each of us through engineering technology; the discovery of new materials of such quality and diversity of properties undreamed of at the beginning of the century; and the development of precise instrumentation and methods of measurement of the properties of these materials that have become the indispensable tools of the materials engineer in evaluating materials for the myriad applications throughout the complex civilization of the world today. These years also have seen two cataclysmic wars, the second of which shook that complex civilization to its very foundations and left social, economic and technologic problems, the solutions of which are the most important things that we face today.

In those fifty years the Society has progressed from small beginnings to a position, we are proud to say, of recognized authority in its field. It has fulfilled the need for a place in which those concerned with the properties and testing of materials may foregather for presentation and critical discussion of the results of their researches, and for arriving at agreement on the provisions to be embodied in standard methods of test and specifications for materials; it has produced standards, for testing and specification of a wide variety of materials, that are indispensable to industry and have proved their value to the Government, especially in times of national emergency; and, most important of all, through the enthusiasm and hard work of its members based upon sound and equitable principles of cooperation, it has developed into an organization ready and capable of assuming the responsibilities that lie ahead in the next fifty years. The full story of A.S.T.M. in the past half century is perhaps best left for the 1952 semicentennial anniversary previously mentioned, but a few observations may be appropriate for this report.

"Small beginnings" have been mentioned, and they were indeed small. Fifty years ago, when we were still the American Section of IATM, there were only 160 members; one committee had been formed (on steel) and there were 10 steel specifications. Today there are nearly 6800 members; 71 technical committees, with their hundreds of subcommittees, are at work with an aggregate membership of about 5500—and this does not include many joint and sectional committees; and as of the close of this annual meeting it is estimated that there will be 1650 standards. Growth has been unspectacular but steady, with the influence of the wars clearly seen in marked stimulation of work in both research and standardization in the post-war periods. Thus, in the 1920–30 decade, standards (which are an excellent measure of activity and accomplishment) increased 160 per cent from 225 to 582; in the 1940–50 decade, 86 per cent from 885 to 1650. These two decades were also periods of steady membership growth: 1600 in the 1920–30 decade and 2400 in the 1940–50 decade. While the depression of the early 30's adversely affected membership, so that there was almost no net growth in the 1930–40 decade, it by no means stopped the growth of our work, standards increasing 52 per cent from 582 to 885 in that period. Other statistics could be quoted "by the yard," showing growth of technical papers, *Proceedings*, special publications, ASTM BULLETIN, and the like, and the development of a sound financial foundation for the Society's work. In the end it would do no more than confirm what the Board believes our members clearly understand, that A.S.T.M. is a hard-working organization that is accomplishing the purposes for which it is organized, is alive to the needs of industry and the technologic professions for "promotion of knowledge of the materials of engineering, and the standardization of specifications and the methods of testing," and is prepared to expand its work within the proper scope of its Charter to grow with the new technologic and industrial developments that have crowded upon the scene since the recent war.

An examination of the history of committee expansion in the five post-war years now ending will illustrate the statement just made. At the beginning of that period there were 57 technical committees; in these five years, 14 new committees have been organized, bringing the total to 71—an expansion of about 25 per cent. This expansion in a *five*-year period is materially larger than the expansion in any previous *ten*-year period, excepting the first decade of the century when the new Society was taking hold of its work and 27 committees were formed. Not all of these new committees represent entirely new work in the Society—in several instances the new committees have taken over lines of activity that were being carried out to some extent in existing committees, such as fatigue, quality control of materials, and appearance standards. For the most part, however, this expansion does represent extension of Society work into fields hitherto not covered, including engine antifreezes, asbestos cement products, magnesium oxychloride and oxysulfate cements, chemical-resistant mortars, structural sandwich constructions, acoustical materials, ceramic whitewares, porcelain enamel, wax polishes, absorption spectroscopy, and methods of testing building constructions. Further evidence of the preparedness of the Society to deal with problems in new fields is seen in the consideration, mentioned in the next section, that is being given to new subjects proposed for study, and in the decision to organize new committees on radioactive isotopes and methods of sampling and analysis for atmospheric pollution. The Board fully recognizes that we are living in a dynamic age with great technologic progress taking place and that the Society must be prepared to move and grow with it.

Merely citing the number of new committees and the subjects undertaken tells only part of the story of expansion in recent years, for considerable expansion has taken place within the framework of existing committees. This is particularly true of a number of the larger committees who have undertaken new projects since the war. No attempt is made to list many items that might be mentioned in this category—they are revealed currently in the reports of the technical committees.

It is significant that the 25 per cent

expansion in technical committees during the post-war period has been accompanied by a 20 per cent growth in Society membership, a 75 per cent increase in assets and nearly 100 per cent increase in surplus,—although with these latter figures it must be remembered that we are dealing with inflated dollars of distinctly less purchasing power than the war-time dollar, let alone the pre-war dollar. There is continued evidence that expansion of technical activities in due time brings its financial support through added income from membership and publications, as it has been doing for many years. Nevertheless, the matter of assurance of adequate financial support for new activities is receiving the attention of the Board of Directors.

The report records briefly the principal happenings and developments of the Society year, including actions by the Board on matters of policy and administration. The growth of technical activities has been stressed and reference made to district and national meetings. The significance and importance of the Society's first national meeting on the Pacific Coast last October is mentioned. Publications, membership, finances, and coöperative activities are covered. At best, the report is but an outline of progress during the year; the full picture appears with the wealth of data, tests, and specifications for this annual meeting and in the publications issued during the year and others in process.

Technical Activities

The annual meeting brings to a close one of the most active years in the technical work of the Society, as will be evident from the committee reports scheduled for the meeting and the record of action on tentatives during the year by the Administrative Committee on Standards. This committee is responsible for following the standardization activities of the Society and its annual report to the Board (see Appendix II) covers some interesting matters. An important function of the committee is to study desirable expansion of our standardization activities and important developments in this direction have stemmed from the committee's recommendations, as noted later.

The report of this administrative committee also notes an important step forward in its efforts for the past few years to improve the quality of A.S.T.M. standards from both editorial and technical points of view, namely, the decision of Committee E-1 on Methods of Testing to prepare a compilation of information useful to all committee members engaged in the writing of A.S.T.M. methods of test, designed to insure more adequate treatment in those methods of such important features as editorial form, concise description of procedures, accuracy of the methods, reproducibility of results, and the like. (Several of the larger technical committees have prepared such information for their own guidance.) Care will of course be taken not to write such "ironclad" rules as would deprive committees of the exercise of judgment and discretion in the writing of methods, especially those of a pioneering nature. It is thought the compilation might take the form of a Manual on the Preparation of A.S.T.M. Methods of Test for distribution to committee officers, and subcommittee and group leaders engaged in this work.

The results of researches carried on by the committees are also revealed in the current reports. Too much emphasis cannot be placed upon the importance of research by our committees, for it is upon the accurate knowledge of properties of materials and methods of determining such properties revealed by research that the quality of the technical content of A.S.T.M. standards, mentioned in the preceding paragraph, must finally rest. The Administrative Committee on Research, whose annual report to the Board appears as Appendix III, keeps in touch with these research activities and lends advice and assistance where desired. The report refers to several large-scale research activities under way or contemplated, such as the research fellowship on X-Ray Diffraction Data at the National Bureau of Standards, the exposure test site program of the Advisory Committee on Corrosion, and the plans for full-size testing of wood poles being developed by Committee D-7 on Wood. Funds for each of these projects are being raised by the respective committees.

The Administrative Committee on Research is continuing the work begun last year of compiling formal statements furnished by the technical committees of unsolved problems in the various technical fields in which A.S.T.M. is at work, for possible publication as an initial listing of research problems for the use of schools, research foundations, and industry.

The brief appended reports of the Administrative Committee on Ultimate Consumer Goods (Appendix V) and Administrative Committee on Simulated Service Testing (Appendix VI) indicate that the committees have in a sense "marked time" for a year in their administrative functions, while considering some of the matters upon which they may become engaged in the future.

New Activities:

A new technical Committee C-22 on Porcelain Enamel was organized at Society headquarters on June 17, 1949, with the following scope:

> The stimulation of research and the formulation of definitions, methods of test and specifications pertaining to porcelain enamels, ceramic and ceramic-metal coatings for metals.

W. N. Harrison has been elected chairman and G. H. Spencer-Strong secretary. The Board's decision to appoint this committee was announced in the report a year ago after conferences with representatives of producers and users of enamels and in consultation with the American Ceramic Society.

A new technical Committee C-3 on Chemical-Resistant Mortars was organized at Society headquarters on October 19, 1949, to take over, on the recommendation of Committee C-12 on Mortars for Unit Masonry, the work of its subcommittee on acid-resistant mortars which had grown to such an extent that it was no longer considered feasible to retain the work within the scope of Committee C-12. The following scope was adopted:

> Formulation of methods of testing, specifications and definitions, and the stimulation of research relating to chemical-resistant mortars.

The work involves a considerable group of test methods and specifications in four main categories, namely, silicate, sulfur, resin, and hydraulic mortars. The following officers have been elected: F. O. Anderegg, chairman, C. R. Payne, vice-chairman, and B. Thomas, secretary.

The Board reported a year ago that it had authorized the formation of a separate technical committee on absorption spectroscopy in view of the decision that hereafter the work of Committee E-2 would be confined to the field of emission spectroscopy. The new committee under the designation Committee E-13 on Absorption Spectroscopy was organized at a meeting in Pittsburgh on February 15, 1950, under the chairmanship of W. R. Brode. A steering committee appointed at that meeting has under consideration such matters as selection of vice-chairman and secretary, determination of scope, completion of personnel, and organization of subcommittees.

During the year the Administrative Committee on Standards recommended to the Board that consideration be given to undertaking work in the Society on floor waxes. Conferences with a number of informed people clearly showed that there is much interest in this subject and that useful work can be done by the Society in the development of methods and specifications as well as in stimulation of research. It seemed desirable, however, to cover the broader field of wax polishes and related materials even though work initially might be centered on floor waxes. Accordingly there was organized at Society headquarters on April 14, 1950, a new technical Committee D-21 on Floor Waxes and Related Materials with the following scope:

> The stimulation of research, and the formulation of test methods, definitions of terms and specifications of wax polishes and related materials, including the materials entering into their manufacture.

The personnel initially has been limited principally to those interested in floor waxes and will be suitably expanded when work is undertaken on other waxes and raw materials. Temporary officers were elected as follows: J. I. Hoffman, chairman, J. Vernon Steinle, vice-chairman, and B. S. Johnson, secretary.

The Marburg Lecture in 1948 by Dr. Paul C. Aebersold of the Atomic Energy Commission at Oak Ridge on the subject "Isotopes and Their Application in the Field of Industrial Materials" aroused considerable interest in the application of such materials to a variety of techniques of testing. As a result of this interest a conference on the subject was held in Pittsburgh on March 1, 1950, under the leadership of Dr. George Manov of the Atomic Energy Commission, in which representatives of the committees of the Society participated, and at which a number of actual uses now being made of radioactive isotopes in the fields of testing covered by the Society were cited. The conference recommended to the Board that the Society recognize the importance of this field by appointing a new technical committee to work in it. The Board concurred and has authorized the President and Executive Secretary to bring about the organization of such a Technical Committee on Radioactive Isotopes.

In recent years there has been marked stimulation in the study by many industries and by various governmental regulatory bodies of problems growing out of pollution of the atmosphere. A most informative discussion of the subject was presented to the Society at a meeting at Franklin Institute on March 8, 1950, under the sponsorship of the Philadelphia District. Consideration of the subject in the Executive Committee of the Board led to the thought that those who have responsibility for the regulation of such pollution would be greatly helped if there were available to them recognized standard methods of sampling and analysis applicable to the various types of atmospheric pollution. Discussions of this idea with various authorities have confirmed the thought that the Society can render a valued service to all concerned if it will engage in the formulation of authoritative methods of sampling and determination of kind and amount of polluting constituents. Accordingly the Board of Directors has authorized the formation of a new Technical Committee on Sampling and Analysis for Atmospheric Pollution and has authorized the President and Executive Secretary to proceed with its organization. In so doing it was expressly stated that the Society would limit its work to the formulation of methods of sampling and analysis and would not concern itself with the problems of specifying limits of pollution nor with other matters concerning biological and health problems.

The subject of examination of metallic substances by electron microscopic methods lies within the scope of Committee E-4 on Metallography. Some months ago the Society was approached by a group of electron microscopists who had formed a Joint Committee on Electron Microstructure of Steel with the proposal that this group, which had thus far been unaffiliated with any organization, would like to consider affiliation with A.S.T.M. The joint committee represented ten or so industrial companies and research foundations, most of whom were members of the Society, and the personnel consisted of an electron microscopist and a metallurgist from each organization. The matter was taken up with Committee E-4 and as the outcome of a conference between the officers of the two groups it was agreed that the joint committee would as a body become Subcommittee XI of Committee E-4 on Electron Microstructure of Steel and function thereafter under E-4 auspices. The results of studies of this group over a period of two years are embodied in an extensive progress report accompanying the 1950 annual report of Committee E-4.

The Board of Directors has under consideration suggestions for work in other fields which have been made by various members of the Society. One of these is smooth surface organic floor coverings, such as linoleum, asphalt tile, rubber tile, etc.; one very informative conference on this subject has been held. A second is the subject of cellulose and cellulose products which is such a broad field touching on many existing activities of the Society that the Board is consulting with interested technical committees in the consideration of this proposal. A specific suggestion from a member of the Society that work be done in the standardization of methods of test of sodium carboxymethylcellulose (CMC) is being considered in conjunction with the broader proposal. A third subject is that of titanium metal and alloys thereof, concerning which there have been recent extensive researches and suggested industrial applications. The possibility that any work the Society does in this field might preferably be initiated in one of the appropriate metals committees is being considered.

Committee Scopes and Jurisdictions:

Scopes of several of the committees of the Society have been passed upon by the Board and the approved scopes are given here as a matter of official record:

Committee C-2 on Magnesium Oxychloride Cements (revised by addition of italicized words):

> The stimulation of research and the formulation of specifications, methods of testing, and definitions relating to magnesium oxychloride *and oxysulfate* cements.

In view of this addition to the scope, the title of Committee C-2 will now be recorded as Committee C-2 on Magnesium Oxychloride and Oxysulfate Cements, although the committee is giving consideration to the possibility of a more abbreviated title.

Committee C-20 on Acoustical Materials (new):

> Study of the properties of those materials used to absorb air-borne sound, including the stimulation of research and the development of methods of tests, specifications and definitions of terms.

Committee C-21 on Ceramic Whitewares (new):

The stimulation of research and the formulation of definitions, methods of tests and specifications pertaining to ceramic whitewares.

Agreement has been reached between Committees D-1 on Paint, Varnish, Lacquer and Related Products and D-20 on Plastics regarding cooperation in the field of plasticizers and resins, and cross representation between the two committees has been completed. The interest of Committee D-2 on Petroleum Products and Lubricants in the field of plasticizers is being examined.

Conferences between Committees D-9 on Electrical Insulating Materials, D-11 on Rubber and Rubber-like Materials, and D-20 on Plastics have been held regarding their common interest in specifications and tests for vinyl plastic materials. Through cross representation now existing in the personnel of these three committees it is believed that the committees can cooperate effectively in the preparation of standards for these materials.

The product known as vulcanized fiber has proved to be of interest to three committees in the Society, namely, D-6 on Paper and Paper Products, D-9 on Electrical Insulating Materials and D-20 on Plastics. Conferences between these committees and with members of the staff have been held with the result that the question of jurisdiction of standards dealing with this product has been determined along the following lines:

1. Committee D-6 is interested in sheet material under 0.030 in. in thickness. It will continue to prepare methods of test applying to paper as such but which might be applicable to thin sheet vulcanized fiber material, and it would be expected that these methods would be made use of by other committees such as Committee D-9 whenever appropriate.

2. Committee D-20 will prepare general methods of testing and specifications applicable to plastics which normally will include vulcanized fiber. Committee D-9, however, will have jurisdiction over any methods of test for electrical properties and joint jurisdiction with Committee D-20 for specifications which include electrical properties. In other words, Committees D-9 and D-20 would have joint jurisdiction over specifications covering vulcanized fiber for electrical uses and would cooperate in the same manner as they now function with respect to a number of standards of interest to both committees.

3. Reference to vulcanized fiber should be removed from methods under the jurisdiction of Committee D-6; at the moment this relates only to the Methods of Test for Ply Adhesion of Paper or Vulcanized Fiber (D 825 - 48). The Method of Test for Bond Strength of Plastics and Electrical Insulating Materials (D 952 - 48 T), prepared by Committee D-20, should be recognized as the method applying to ply adhesion of vulcanized fiber.

District and Related Activities

As noted in the Report of the Administrative Committee on District Activities which is appended (Appendix VII), the various A.S.T.M. Districts functioned effectively under the current Charter and Manual, and no major administrative matters arose during the year. The annual election of councilors whose terms expire this year is under way, and the biennial election of officers in each of the Districts (Chairman, Vice-Chairman, and Secretary) is being conducted in line with the regulations which provide for the appointment of a Nominating Committee in the District, and then a letter ballot of the respective District memberships. The July ASTM BULLETIN will carry a complete list of new officers and new or reelected councilors.

During the year the Ohio Valley District, centered in the Ohio, Indiana, Kentucky areas, was formally organized and has already planned a program of meetings. The thirteen Districts now organized blanket the northcentral and northern and eastern United States, and

California, and include at least 80 per cent of the members in the United States and Canada.

As noted in the Administrative Committee Report, several Districts have aided with national meetings. The Northern and Southern California District Councils were a nucleus for the General Committee on Arrangements for the Pacific Area National Meeting; the Philadelphia District was the Host Group for the 1949 Annual Meeting and is repeating for 1950; and the Pittsburgh District arranged a reception for President Morrow during Committee Week in March, 1950. The Society greatly appreciates the splendid cooperation of the Districts in connection with national meetings.

District Meetings:

The accompanying table lists all of the A.S.T.M. National and District Meetings during the year, with notes on the features of various District meetings. Many of these meetings were sponsored jointly with local sections of other organizations, and it is expected this will continue to be the case because of its advantages.

A number of the District-sponsored meetings had very interesting programs and excellent attendance. A list of these would include the one in Detroit with its several papers on Applying Man-Made Isotopes in Industry; the New York meeting on Isotopes in Industry; the Philadelphia meeting jointly with the Franklin Institute, on Research for Management with several papers at afternoon and evening sessions and a dinner between sessions. The attendance was excellent at two other Philadelphia District gatherings: one on Stress Analysis, which was under the joint auspices of the Philadelphia District and the local section of the Society for Experimental Stress Analysis, during the January meetings of Committee A-1 on Steel; and the other, a discussion of Atmospheric Pollution, a most interesting and timely subject.

The Western New York-Ontario District was pleased to cooperate with the Niagara Peninsula Branch of the Engineering Institute of Canada for its meeting on April 21, in St. Catharines, Ontario, and to have as its guest from his "Home District," President J. G. Morrow of Hamilton, Ont.

Despite a heavy schedule due to the demands of his business and also his responsibilities as Chairman of the Canadian Standards Association, President Morrow was able to attend a number of District meetings, and spoke briefly at each. The Executive Secretary or one of his staff associates attended nearly all these meetings, usually speaking briefly about some phase of the Society's work.

Contacts with Members in West and Southwest:

In October while the Pacific Area Meeting was in progress, a number of officers of the Society and staff members were able to meet for the first time many members who do not find it possible, as a general rule, to attend Eastern or Midwestern Society meetings. In March, to follow through some of these contacts, and also to get our members together in certain other areas, the Assistant Secretary made an extensive Western trip. Dinner meetings were held in the Twin Cities (Minneapolis-St. Paul), in Portland, and in San Francisco and Los Angeles with the Northern and Southern California Districts. Informal conferences were held with members in Seattle, and in Dallas, Houston, and New Orleans and short luncheon sessions took place. These informal affairs were the first opportunity (other than in California) that our members in these areas have had to get together, and in each instance the groups expressed the hope that periodically some officer of the Society or staff member can meet with them and discuss pertinent A.S.T.M. activities which may be of particular concern to the respective areas.

It is apparent that the tremendous growth of industry in many sections west of the Mississippi has not been fully reflected in growth of the Society membership, although there is a steady acquisition of new members from these sections. With the cooperation and influence of groups of our people in the various sections, interest and participation in the work of the Society can and should be more actively stimulated.

National Meetings

The 1949 Annual Meeting, the Society's Fifty-Second, held in Atlantic City, was marked by a very diversified technical program and upwards of 450 meetings of technical committees, the largest number ever held at a national meeting of the Society. The attendance of 1857, the second highest, indicates the intensive interest in the features of the meeting. In order to provide an appropriate opportunity for the presentation of the President's Annual Address, introduction of new officers, and recognition of long-time members, there was held for the first time a luncheon session. This worked out so well that at this 1950 meeting, with more events of this kind, including Honorary Memberships and the first Awards of Merit, two luncheon sessions have been scheduled.

A very interesting entertainment program for the ladies was provided by the Philadelphia District, hosts for the meeting, who also sponsored an excellent dinner and entertainment.

From February 27 to March 3, 1950, there was held the annual Committee Week in Pittsburgh at which nearly 1000 members and visitors were registered to attend about 250 committee and subcommittee meetings. No technical session to constitute a Spring Meeting was arranged.

Pacific Area National Meeting:

From October 10 to 14, 1949, the First Pacific Area National Meeting was held in San Francisco, with headquarters at the Fairmont and Mark Hopkins Hotels. There were 69 technical papers presented in fifteen sessions, and approximately 65 meetings of technical committees were held. Several well-attended industry luncheons were held, and there were various organized trips including an all-day trip to the University of California. The meeting was also marked by a very interesting entertainment program. The registered attendance was about 700 men and 110 ladies, but it is estimated that more than 1000 men were at the meeting sometime during the week, at technical sessions, and committee meetings.

The meeting was an excellent one in every respect. Many of the technical contributions, including symposiums as well as individual papers, are being published, and announcements appear in the A.S.T.M. BULLETIN on the availability of the papers. Not only did the meeting provide an opportunity for many of our eastern members (more than 25 per cent of the attendance was from east of the Mississippi) and officers to meet with members and visitors from the eleven Pacific Area states, which was a most

A.S.T.M. MEETINGS, JUNE 1949–MAY 1950, INCLUSIVE.

Meeting	Place and Date	Notes
Annual Meeting	Atlantic City, N. J. Hotel Chalfonte-Haddon Hall June 27–July 1, 1949	22 Technical Sessions; Several Informal Round Table Discussions, and Groups of Papers 450 Committee Meetings (Registered Attendance 1857—335 Ladies)
Pacific Area National Meeting	San Francisco, Calif. Fairmont Hotel October 10–14, 1949	15 Technical Sessions[a] 65 Committee Meetings (Registered Attendance about 700—total attendance at meeting over 1000, including 113 ladies.)
New York Meeting	New York City, N. Y. Biltmore Hotel December 28, 1949	Joint Meeting with American Statistical Assn. during the latters' Annual Meeting 2 Technical Papers on Statistical Design of Enigneering Tests by Grant Wernimont, and W. J. Youden.[b]
A.S.T.M. Committee Week	Pittsburgh, Pa. Hotel William Penn February 27–March 3, 1950	260 Committee Meetings (About 1000 in Attendance) President's Reception
District or Local (sponsored by Councils indicated)	Philadelphia, Pa. Franklin Institute October 12, 1949	Jointly with Philadelphia Section A.S.Q.C. Quality Control in Materials Procurement, by Robert Burns.
	Boston, Mass. (New England) Northeastern University October 24, 1949	Solar Heating: The Architectural Aspects, by L. B. Anderson; The Engineering Considerations, by A. G. H. Dietz
	Detroit, Mich. Rackham Memorial Bldg. October 27, 1949	Applying Man-Made Isotopes in Industry: Significant and Historical Backgrounds on Isotopes, by T. M. Switz; Tracer Elements in the Inorganic Field, by Harrison Brown; Tracer Elements in the Organic Field, by Weldon G. Brown; Cautions and Precautions in the Use of Radioactive Isotopes, by Wendell C. Peacock.
	New York, N. Y. Engineering Societies Bldg. October 28, 1949	Jointly with New York Section A.S.M.E. Isotopes in Industry, by John R. Dunning; Future of Nuclear Power, by Albert L. Baker
	St. Louis, Mo. Engineers Club November 3, 1949	Jointly with Engineers Club of St. Louis Major Sources of Energy, by Eugene Ayres
	Buffalo, N. Y. (Western New York-Ontario) Park Lane Restaurant November 7, 1949	Smoker: Remarks by J. G. Morrow and C. L. Warwick
	Pittsburgh, Pa. Mellon Institute November 29, 1949	Quality and Utility in Modern Glass, by Alexander Silverman
	Philadelphia, Pa. Franklin Institute December 14, 1949	Jointly with Franklin Institute Research for Management: The Private or Consulting Research Laboratory, by Milton Harris; The Colleges and Universities, by Harold K. Schilling; The Non-Profit Institutes, by Edward R. Weidlein; The Importance of Research, by Vice-Admiral Howard G. Bowen; Some Aspects of the Function, Organization, and Operation of Industrial Research and Development Laboratories, by Dudley E. Chambers; Research from the Standpoint of the Smaller Business Enterprise, by G. H. Clamer; Summary of the Day's Proceedings by W. F. G. Swann.
	Philadelphia, Pa. Benjamin Franklin Hotel January 31, 1950	Jointly with Philadelphia Section, S.E.S.A. Stress Analysis in Action, by Wm. T. Bean, Jr.
	Philadelphia, Pa. Franklin Institute March 8, 1950	Atmospheric Pollution, by Louis C. McCabe; Atmospheric Pollution and Pressure to Abate It, by W. B. Hart
	Los Angeles, Calif. (Southern California) Rodger Young Auditorium March 23, 1950	A.S.T.M. Activities Brought Up to Date, by Messrs. Painter, Lake, Stringfield, Kelch, Delmonte, and Bolte
	New York, N. Y. Engineering Societies Bldg. March 24, 1950	Jointly with N. Y. Section, A.S.M.E. Machine Design Division. Electronics Research for the Department of National Defense, by Edwin A. Speakman

A.S.T.M. MEETINGS, JUNE 1949–MAY 1950, INCLUSIVE (*Continued*)

Meeting	Place and Date	Notes
District or Local (sponsored by Councils indicated)	St. Louis, Mo. Engineers Club March 28, 1950	Jointly with Engineers Club of St. Louis Stress Analysis in Action, by Wm. T. Bean, Jr.
	Chicago, Ill. W.S.E. Headquarters March 29, 1950	Modern Photography in Industry:—Eastman Kodak Film, "Functional Photography in Industry," comments by John Niemeyer
	Boston, Mass. (New England) Northeastern University April 10, 1950	Competitive Sources of Energy, by Warren K. Lewis
	St. Catharines, Ont., Canada (Western New York-Ontario) Hotel Queensway April 21, 1950	Jointly with Niagara Peninsula Branch, E.I.C. Functional Photography: Eastman Kodak Film, "Functional Photography in Industry," comments by George Eaton; Plant visits in afternoon
	New York, N. Y. Port Authority Auditorium May 11, 1950	Tunnel and Terminal—How They Were Built, Tunnel, by Ralph Smillie; Terminal, by John M. Kyle
	Welland, Ont., Canada (Western New York-Ontario) Barclay Hotel May 18, 1950	Jointly with Niagara Peninsula Branch, E.I.C. Inspection Trip and Technical Meeting on Large Pipe for Oil Fields; Talk by C. W. Moorehead

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[a] Many of the Symposiums and individual papers are being published, and details have been given in ASTM BULLETINS.

[b] Published in ASTM BULLETIN No. 166, May, 1950, p. 45.

valuable function of the meeting, but the meeting brought about a much wider knowledge of the Society's work throughout the far west. It further proved to be a truly integrating force such as we have never had before.

The December, 1949, ASTM BULLETIN carried a full account of the meeting, with notes on the technical sessions and committee meetings. The complete technical program has also been recorded in the 1949 *Proceedings*. Too much credit cannot be given the General Committee on Arrangements which aided in developing the technical program, planned the entertainment and social features, raised a substantial sum of money to underwrite costs of meeting rooms and many of the social events, and in many other ways contributed to the smooth and effective conduct of the meeting. At its January meeting, the Board of Directors adopted a formal resolution of thanks to the General Committee on Arrangements and to the members of all its committees for their services to the Society.

Joint Meeting with the American Statistical Association:

The Society joined with the American Statistical Association in a session on Statistical Design of Engineering Tests, which was held as a part of the annual meeting of that Association in New York on December 28, 1949. The two papers presented at the session together with the discussion brought out considerable information of value in the application of statistics to engineering tests. These papers were published in the May, 1950, issue of the ASTM BULLETIN. The Executive Secretary acted as chairman of the session.

1951 and 1952 Meetings:

The 1951 Annual Meeting, the Society's Fifty-Fourth, will be held at Chalfonte-Haddon Hall, Atlantic City, throughout the week of June 18.

The 1951 Committee Week and Spring Meeting will be held at the Netherlands-Plaza Hotel in Cincinnati during the week of March 5.

In 1952, the Society will celebrate the Fiftieth Anniversary of its incorporation as a national technical society, and a number of special events are being planned for the Annual Meeting that year, which will be held at the Statler Hotel in New York during the week of June 22. The biennial Apparatus and Photographic Exhibits will be held in conjunction with the meeting.

The 1952 Committee Week and Spring Meeting will be held in Cleveland during either the week of February 25 or the week of March 3.

Publications

Responsibility for the publications of the Society is delegated to the Administrative Committee on Papers and Publications. The report of that committee is attached as Appendix IV; it reviews the activities of the committee and gives interesting information about the publications for the past year as well as some future publications.

The annual volume of the *Proceedings* (1252 pages) and the six parts of the Book of Standards (approximately 8400 pages) were the major 1949 publications. This is the first time the Standards have been published in six parts. The magnitude of these publications constituted a heavy load on both the editorial staff and the printing establishment, and the final book was not completed until May, 1950. Twenty-two compilations of standards were printed and together with various special publications listed in the Committee's report comprise an important part of the 1949 publications.

In the mid-year the ASTM BULLETIN was placed on an eight-issue basis, so that seven issues were published in 1949 comprising a total of 662 pages, made up of 239 pages of technical papers and discussions, 256 pages of news about Society activities and 167 pages of advertising (not including cover advertising). The average edition was nearly 10,000.

Of the publications in hand for 1950, preprints of reports and papers for this annual meeting are now being distributed to the members. *Proceedings* and the 1950 Supplements to the six parts of the Book of Standards, as well as an edition of the Book of A.S.T.M. Methods of Chemical Analysis, will be taken up in the summer and fall. Much has already been accomplished in editing and printing many of the papers presented at the Pacific Area National Meeting last October. Some are appearing currently in the BULLETIN; others will be included in the 1950 *Proceedings;* still others are being grouped into six Special Technical Publications as noted in the committee's report, with several more to be gotten under way.

Publication by the Society, for the Joint Committee on Chemical Analysis by X-Ray Diffraction, of 1000 sets of the X-Ray Diffraction Cards—which when completed will number over 4000—is one of the biggest undertakings of the publication schedule. For the first time some sets of these cards have been punched for use of the "key sort" method of classification. An index of the cards is an important part of the publication.

Another important joint publication is under way, namely, a Manual of Oil Measurement Tables being compiled by the Society (through Committee D-2 on Petroleum Products and Lubricants) and the British Institute of Petroleum. Computation of the tables has been completed on IBM machines and is now being checked. Methods of publication are being discussed with the Institute, and work on the final editing and printing may be begun before the close of the year. There are 37 tables in all, covering U. S., British and Metric units, which will comprise close to 1100 pages.

Attention is directed to the excellent work by the Administrative Committee on Papers and Publications in preparing a Manual on the Preparation of Technical Papers (part of which was published in the April, 1950, BULLETIN) and an article on "ASTM Papers—Their Preparation, Acceptance and Publication" which appeared in the January, 1950, BULLETIN. When finally issued, these will constitute a most valuable reference for authors of papers and discussions and an aid to the editorial staff.

Administrative Matters

Regulations Governing Technical Committees:

Three revisions in the Regulations Governing Technical Committees[1] have been approved by the Board of Directors and made effective by announcement to the technical committees and by publication in the BULLETIN. They are included here for the official record with brief comments.

Numeric Balance of Members of Technical Committees.—The present provision in Article I, Section 2 (*g*) of the Regulations concerning balance between producer, consumer and general interests has been changed to read as follows:

> On such committees either an equal numeric balance shall be maintained between the producers on the one hand and the consumers and general interests, jointly, on the other hand; or the consumers and general interests may be allowed to predominate in the absence of objection by a majority of the producers. Thus a substantial balance may be permitted to exist between the three general classifications of members on technical committees.

The reasons for this change were explained in the Board's report a year ago when the proposed revised wording was published as being under consideration and awaiting comments from the technical committee chairmen. After considering all such comments, the Board affirmed its previous action.

Letter Ballots of Technical Committees on Recommendations Affecting Standards.—Discussion of the preceding change emphasized the importance and significance of the letter ballots of committees in acting upon recommendations affecting standards, especially in relation to the matter of numeric balance. It was decided to propose to the technical committee chairmen that, referring to the provisions of Article III, Section 14 (*a*) of the Regulations, the letter ballot votes of producers on the one hand and of consumers and general interests on the other hand be counted separately, and that an affirmative vote of two-thirds of those voting *in each of the two groups* shall be required for adoption of recommendations affecting standards, instead as heretofore of the total number voting irrespective of classification of members. Thus, a recommendation will fail of passing if more than one-third of *either* producers on the one hand or consumers and general interests on the other hand oppose it, which gives a further protection to a minority of either group. No modification to this proposed change in the light of comments received from the technical committee chairmen seemed necessary, and it was accordingly adopted. Following is the revised wording of Section 14 (*a*):

> 14. Procedure Governing Recommendations on Standards.—(*a*) Committee recommenda-

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[1] 1949 A.S.T.M. Year Book, p. 534.

tions affecting standards shall normally be agreed upon at regularly called meetings. (For an alternate to this "normal" procedure, see Paragraph (*b*).) An affirmative vote amounting to two-thirds of those voting shall be required on all recommendations affecting standards, subject to confirmation by letter ballot of the entire committee on which an affirmative vote amounting to two-thirds of the combined number of consumers and general interests voting and two-thirds of the producers voting shall also be required. The number of affirmative, negative, and "not voting" ballots shall be included in the report of the committee to the Society. If the number of negative votes on the letter ballot exceeds 10 per cent of the sum of the affirmative and negative votes, the report to the Society shall also contain a classification of the total vote as to producers, consumers, and general interests (see Section 2 (*h*)).

Special Procedure for Writing Ultimate Consumer Goods Standards.—In its report a year ago, the Board presented proposed modifications of the Regulations to cover the development of standards in the ultimate consumer goods fields wherever it is not feasible to establish in the entire technical committee the balance of interests between producers, consumers and general interests prescribed in the regular procedure. It is the intention that any work on standards for the ultimate consumer shall be handled within the framework of our present committee organization, but special provision is made for having consideration of such standards in representative subcommittees. Comments from technical committee chairmen were generally favorable, but one committee proposed an alternative wording to which the Board gave consideration during the year. It was decided to adhere to the wording proposed, and the revision was finally adopted and announced to the technical committees. The revision consists of the following, the new Section 14 (*c*) comprising the essence of the revision:

1. Insert at the end of Article I, Section 2(*g*):

> An exception to this rule may be involved in certain actions connected with ultimate consumer goods as set forth in Article III, Section 14 (*c*).

2. Add the following to Article II, Section 9:

> (See also Article III, Section 14 (*c*)).

3. Change Article III, Section 14 (*c*) to Section 14 (*d*).

4. Insert the following new Section 14 (*c*):

> 14 (*c*). In certain cases of actions involving ultimate consumer goods it may not be feasible to establish in the appropriate existing technical committee the balance of interests concerning a particular item of consumer goods as prescribed in Article I, Section 2 (*g*). In such cases where recommendations regarding standards or recommended practices involve ultimate consumer goods, the following special provisions shall apply. The technical committee may refer the matter with proper recommendations to a special subcommittee which shall consist of not fewer than nine members and to which shall be applied (rather than to the technical committee) the balance of interests as prescribed in Section 2 (*g*) including representation of the ultimate consumer. Recommended action by such a subcommittee shall be by not less than two-thirds affirmative vote by letter ballot. Such subcommittee recommendations shall be addressed to the technical committee and shall then be handled as usual under Sections 14 (*a*) or (*b*).

Procedures of Administrative Committee on Standards:

The Board of Directors considered a proposal from one of the committees of the Society that the By-laws be amended to include a description of the procedure to be followed by the Administrative Committee on Standards in considering negative votes on recommendations that come before it. An examination of the question in consultation with the Administrative Committee on Standards led the Board to the conclusion that it is not advisable to include such procedure in the By-laws, and that the point raised could be handled satisfactorily by having the

Administrative Committee on Standards incorporate in its operating regulations a description of its procedure with respect to the privilege of a member voting negatively on recommendations coming before the Administrative Committee of being present at meetings to support his viewpoint, or of amplifying his point of view if the recommendation is being acted upon by letter vote of the Administrative Committee. It was decided that if such member voting in the negative felt that his case is of sufficient importance, he should have the privilege of petitioning the Administrative Committee for a formal hearing before action is taken. Accordingly the Board has directed the Administrative Committee on Standards to incorporate such provisions in its operating regulations and to continue its practice of informing committee officers of such provisions.

Headquarters Staff:

The Society and its staff suffered a grevious loss in the death, on November 17, 1949, of Carter S. Cole, Assistant Technical Secretary. The Board adopted the following resolution at its January, 1950, meeting:

> It is with profound sorrow that the Board of Directors records the untimely death, on November 17, 1949, of Carter Stanard Cole, Assistant Technical Secretary of the Society. While officially on the Headquarter's staff only some five years, he had long been active in the Society and its committees, which enabled him to bring to his duties as a staff member a wealth of experience and judgment that gained the respect of his colleagues and committee associates. He was particularly interested and helpful in procedural matters, and his advice in these was frequently sought.
>
> But he was particularly esteemed for the willing and cooperative spirit with which he entered upon all his assignments and which entered into all his associations, and his presence will be sorely missed. The Board, in adopting this resolution as a tribute to him, extends its sympathy to his family and his colleagues.

Two members of the staff were recognized by the Board through suitable citations which have been published in the BULLETIN, namely, John K. Rittenhouse, Treasurer, who completed 40 years of service on October 9, 1949; and George A. Wilson, Assistant Editor, who completed 25 years of service on December 15, 1949.

In August, 1949, William F. Black, Jr., was engaged in the editorial department of the staff, completing a plan of expansion started two years ago. In April, 1950, Sheldon E. Fitterer was added to the staff of the standards department, restoring the technical and administrative staff after Mr. Cole's death to its authorized complement of fourteen. Notes regarding each of these new staff men have appeared in the BULLETIN. Recently an additional stenographer was engaged for the technical and editorial departments, bringing the total headquarters staff to 54, a net increase of two during the year. In addition, several clerks and stenographers are employed on a part-time basis to meet peak load conditions.

Society Headquarters:

The headquarters have continued to find favor with the members of the Society and its facilities have been used by both committees and members. Thus, during the calendar year 1949 there were 76 committee meetings and conferences held in the building, including several meetings of other organizations to whom the Society is always pleased to extend this courtesy.

During the year an air-conditioning unit was purchased for the fourth floor at a cost of $440.75, and the soundproofing of the office space requiring it was completed at a cost of $1432,* each of these being a capital charge against the Building Fund.

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* Only $386.00 of this amount appears in the auditors' report for fiscal year 1949; the remainder was expended in 1950.

Financial Aspects.—Later in this report, in the section on finances and in the auditors' report covered in Appendix I, is given full iuformation regarding the operations of the Building Fund through which the purchase of the headquarters was made. The headquarters building is carried on the balance sheet as an asset after depreciation of $170,944.87. The building is nearly clear of indebtedness, there being only a note payable to General Funds of $13,866.67 as of December 31, 1949. Provision is made in the general budget under Headquarters Occupancy Expense to pay off this loan at the rate of approximately $1130 a year, but it is hoped that further contributions to the Building Fund will make possible a more rapid reduction of that indebtedness.

The cost of operating the headquarters building in 1949, including full depreciation charges and portions of staff salaries chargeable to management and maintenance service, was about $2.00 per sq. ft.

Membership

On June 1, 1950, the membership of the Society totaled 6761, compared with a total of 6620 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	June 1, 1949	June 1, 1950	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increase
Honorary	18	16	...	...	2	...	...	...	2	...	2[a]
Perpetuity and Life	11	11	...	...	...	...	...	...	...	...	...
Sustaining	217	214	1	...	...	5	1	2	6	3	3[a]
Company, Firm, etc.	1625	1658	66	20	...	21	33	107	107	140	33
Individual, etc.	4627	4739	224	116	43	28	36	487	411	523	112
Junior	122	123	9	5	...	16	...	31	30	31	1
Total	6620	6761	300	141	45	70	70	627	556	697	141
Student	589	493	23	317	...	19	...	273	369	273	96[a]

[a] Net loss.

It will be noted that 627 new members were elected, compared with 626 for the preceding year. However, there was a somewhat greater loss from resignation, delinquency, and death compared with the previous year, so that the net increase of 141 is lower than the net increase of 182 a year ago, although it compares favorably with the net increase of 125 reported two years ago.

It is encouraging that the Society's total membership keeps increasing, but considering the breadth of A.S.T.M. work in standards and research, it is obvious that many more individuals and companies would unquestionably benefit from affiliation with the Society, and conversely that the Society would benefit from the participation in the work of a larger membership.

Sustaining Members:

There have been three new sustaining members acquired, and there were six losses, five of these being transfers to company membership. The three new sustaining members are:

Huron Portland Cement Co.
Procter & Gamble Co.
Republic Steel Corp.

50-Year Members:

There are six individuals and companies as noted below that have been connected with the Society continuously for 50 years, having joined the American Section of the International Association for Testing Materials in 1900:

Theodore L. Condron
Robert Job
American Steel and Wire Co.
Colorado Fuel and Iron Corp.
National Tube Co.
John A. Roebling's Sons Co

These members will be recognized at this Annual Meeting.

40-Year Members:

There is a total of 25 members who this year have completed forty years of membership, which brings the total number of members currently in the "Forty-Year Club" to 153. The members who will receive 40-Year Certificates at this Annual Meeting are as follows:

Lawrence Addicks
American Transit Assn.
Armco Steel Corp.
Charles V. Bacon
P. H. Bates
Bausch & Lomb Optical Co.
L. G. Blackmer
British Ceramic Society
Cementos Mexicanos, SA
Flint C. Elder
General Electric Co., Pittsfield Works
John Howe Hall
A. E. Horn
William H. Klein
Lehigh Valley Railroad Co.
M. E. McDonnell
J. E. Myers
National Crushed Stone Assn., Inc.
City of St. Louis Municipal Testing Laboratory
Stanford University Library
State University of Iowa Libraries
Studebaker Corp.
Western Union Telegraph Co.
Francis P. Witmer
G. H. Woodroffe

Deaths:

The Society has lost the following 45 members through death:

Badger, Irvin S. (1943)[a]
Barr, Joseph R. (1923)
Basch, David (1946)
Billings, A. W. K. (1915)
Cole, Carter S. (1935)
Cromwell, Oliver C. (1906)
Crum, B. M. (1931)
Dickinson, H. C. (1911)
Easley, M. K. (1941)
Evans, Charles T., (1924)
Fulton, A. M. (1930)
Gathmann, Emil (1914)
Gillett, H. W. (1924)
Greensted, H. B., Sr. (1935)
Guyer, Paul B. (1949)
Hall, Kenneth S. (1946)
Harriman, N. F. (1902)
Heath, Fred (1949)
Hicks, Thomas A. (1914)
Holmes, Addison F. (1906)
Jeffries, William J. (1935)
Kennedy, J. C. (1922)
Kilgus, H. E. (1917)
Knight, Burke Haywood (1942)
Lagerqvist, Joel (1947)
Lyon, P. W. (1944)
Mabe, Ralph W. (1944)
Marston, Anson (1905)
Milburn, Henry M. (1917)
Millar, Preston S. (1942)
Miner, Joshua L. (1912)
Morehead, F. H. (1926)
Mort, Linwood G. (1946)
Nichols, Ernest H. (1933)
Olson, H. M. (1941)
Osborn, Kenneth H. (1927)
Price, William B. (1913)
Randall, Merle (1944)
Reeve, C. S. (1904)
Rexford, Elliot P. (1946)
Schley, Charles E. (1924)
Schwartz, Joseph L. (1945)
Smith, Arthur G. (1940)
Tolman, L. M., (1936)
Young, Edward R. (1933)

In addition to the above, the following representatives of company members of the Society passed away:

L. S. Converse, American Viscose Corp.
P. Diederich, City of Glendale, Calif., Public Service Dept.
E. R. Dillehay, The Richardson Co.
Joseph C. Dooley, New York Trap Rock Corp.
F. A. Hartman, Protexol Corp.

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[a] Date of membership.

R. W. H. Hawken, University of Queensland, Faculty of Engineering.
Harry D. Jumper, Consolidated Rock Products Co.
W. W. Knight, Roth Rubber Co.
Gerald T. Kurtz, The Paraffine Cos., Inc.
N. L. Marks, Jr., New Orleans City Engineer's Office.
Ray P. Mattern, Minneapolis-Honeywell Regulator Co.
Frederick Mills, Western Australian Government Railways.
A. C. Nixon, Fisher Body Detroit Division, General Motors Corp.
Burton Paxton, Chicago Hydraulic Oil Co.
J. R. Putnam, The United States Time Corp.
John Jay Shank, The Wayne Laboratories.
A. G. Tainton, Pretoria Portland Cement Co.
James Weir, The James Weir Co., Ltd.
R. J. Wheeler, Carter Traveler Co.

A number of these men have been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The notable work and meritorious service of two of the men, W. B. Price and C. S. Reeve, had been recognized through election to Honorary Membership in the Society. Both had served as members of the Board of Directors. Several others had notable records of accomplishment for the Society. Mr. Basch had for many years, as a committee representative of the General Electric Co., been active in Committee B-6 on Die-Cast Metals and Alloys. Mr. Cromwell, retired for many years, had a long period of service in Committee A-1 on Steel. H. C. Dickinson, long with the National Bureau of Standards, was active on Committee D-2 on Petroleum Products and Lubricants. H. W. Gillett, an outstanding authority in the metallurgical field and first Director of Battelle Memorial Institute, gave unstintingly of his time and wide knowledge to the Society; many of the Society's technical publications bear the stamp of his facile pen. N. F. Harriman, for many years vice-chairman of the Federal Specifications Board and previously with the Union Pacific Railroad, served on several committees in the Society's early years. A number of other men had been concerned with technical committee work: T. A. Hicks particularly in C-1 on Cement where he was an honorary member; W. J. Jeffries, formerly with the U. S. Navy, on Committee A-1 on Steel; H. M. Milburn, for many years with Committee D-4 on Road and Paving Materials. F. H. Morehead for many years represented the Manufacturers Standardization Society of the Valve and Fittings Industry on Committee A-1 on Steel and other ferrous groups, and E. R. Young served on Committee A-3 on Cast Iron, of which he had been secretary and chairman, and also on A-1 on Steel. E. H. Nichols was a member of several committees in the building materials group. F. A. Hartman was an active member of Committee E-5 on Fire Tests of Materials and Construction; H. D. Jumper contributed of his time and effort particularly to Committee C-9 on Concrete and Concrete Aggregates; and R. J. Wheeler was connected with the work of Committee B-5 on Copper and Copper Alloys.

Special mention should be made of the long-time interest of Dean Anson Marston who had served as a Director of the Society and for many years headed Committee C-4 on Clay Pipe. J. L. Miner devoted much effort to the Society, especially in Committee C-1 on Cement; he also had served on the Board of Directors and at the time of his death was a member of the Administrative Committee on Standards.

Honors and Awards

Periodically the Board of Directors acting for the Society takes cognizance of outstanding service through election to Honorary Membership, and the recent establishment of the Award of Merit by the Board provides a further means of recognizing important service to the Society. Various awards and medals are also given to recognize outstanding papers and to foster research in certain special fields of activity.

Honorary Membership:

During this annual meeting the following six members, whom the Board recently elected to Honorary Membership, will be presented to the Society for their formal awards; each has over long periods of years contributed greatly to the advancement of the Society's work:

Phaon H. Bates (retired), formerly Chief, Clay and Silicate Products Division, National Bureau of Standards, and Past President of the Society.

Wilson C. Hanna, Chief Chemist, California Portland Cement Co., and Past Director of the Society.

Dean Harvey (retired), formerly Materials Engineer, Westinghouse Electric Corp., and Past President of the Society.

Prévost Hubbard (retired), formerly Chemical Engineer, Asphalt Institute, and Past Director of the Society.

Robert Job (retired), formerly Vice-President, Milton Hersey Co., Ltd., and Past Director of the Society.

H. S. Rawdon (retired), formerly, Chief, Division of Metallurgy, National Bureau of Standards.

Award of Merit:

As announced a year ago the Board of Directors has established an A.S.T.M. Award of Merit as a further means of recognizing individuals who have rendered distinguished service to the Society. The Award of Merit Committee for 1950, comprising T. A. Boyd, chairman, D. E. Parsons, C. H. Rose, H. S. Vassar, and L. H. Winkler, reviewed numerous suggestions and citations from the various technical committees and nominated nine members who by unanimous action of the Board were voted the 1950 Awards of Merit. Each one has contributed in an important way to the work of the Society in various of our technical fields. They will be presented to the Society during this annual meeting to receive the Award of Merit certificates. They are as follows:

Alfred P. S. Bellis
Allan W. Dow
Daniel W. Kessler
T. Smith Taylor
LeRoy L. Wyman
Harold F. Dodge
Howard P. Ferguson
Thomas G. Stitt
Percy H. Walker

Recognition of Technical Papers:

In recognition of certain outstanding technical papers, three awards will be made at this annual meeting as follows:

Charles B. Dudley Medal—presented for a paper of outstanding merit constituting an original contribution on research in engineering materials. This year it will be presented to Prof. B. J. Lazan, Syracuse University, for his paper entitled "Dynamic Creep and Creep-Rupture Properties of Temperature-Resistant Materials under Tensile Fatigue Loading."

Richard L. Templin Award—presented for a paper describing new testing methods and apparatus, the purpose of the award being to stimulate research in the development of testing methods and apparatus. This year it will be given to Professors D. S. Clark and D. S. Wood, California Institute of Technology, for their paper entitled "The Time Delay for the Initiation of Plastic Deformation at Rapidly Applied Constant Stress."

Sam Tour Award—presented for the purpose of encouraging research on the improvements and evaluation of corrosion testing methods and to stimulate the preparation of technical papers in this field. This year the award will be made to O. B. Ellis, Armco Steel Corp., for his paper entitled

"Effect of Weather on the Initial Corrosion Rate of Sheet Zinc."

The Sanford E. Thompson Award, which recognizes papers of outstanding merit in the field of concrete and concrete aggregates, will not be awarded this year.

The Harold DeWitt Smith Memorial Medal was presented to Herbert F. Schiefer of the National Bureau of Standards at the March 16, 1950, meeting of Committee D-13 on Textile Materials, in recognition of his work in the science of utilization of textile fibers.

Finances

Report for the Fiscal Year of 1949:

The annual statement of the finances of the Society is presented in the form of the report of the auditors for the fiscal year January 1 to December 31, 1949, which appears in Appendix I. The report gives the balance sheet of assets and liabilities, including general funds, building fund, and other special and designated funds; a statement of receipts and disbursements classified into "operating" (budgeted) and "nonoperating"; details of the building fund and of special funds; and investments of Society funds.

Total operating receipts were $502,047.52, the second largest in the Society's history. Of this amount receipts from dues and entrance fees were $202,781.66 or 40.4 per cent; receipts from sales of publications were $253,126.94 or 50.4 per cent; receipts from miscellaneous sources consisting principally of advertising, interest and dividends, and registration fees, total $46,138.92 or 9.2 per cent. Receipts from publication sales were particularly gratifying and reflect strong demand for the Book of Standards and Supplements and for the various compilations of standards. It is significant that approximately 81 per cent of publication sales are accounted for by publications growing out of our standardization work, the remaining 19 per cent comprising sales of *Proceedings*, technical papers, BULLETIN, and various special and miscellaneous publications. This shows strikingly the value of the Society's publications as a major source of operating income.

Operating disbursements with all current bills paid (as detailed in the auditors' report) were $490,871.43. Of this amount $205,079.73 was spent for publications and $172,731.95 for salaries, comprising 76 per cent of the total. (This ratio has varied over the past decade from about 74 to 82 per cent, depending largely upon volume of annual publications.) The remaining disbursements of $113,059.75 were divided among items of general office expense, expense of technical committees and district councils, meetings, headquarters occupancy (equivalent rent), employees retirement fund, and miscellaneous items.

The payment into the principal of the A.S.T.M. Research Fund of one-half of the current entrance fees of about $2500 was deferred for the third successive year, the Board feeling that the monies involved are needed for and can be used to better advantage in support of expanding technical activities, including the expenses involved in the organization of new technical committees.

The difference between operating receipts and disbursements constitutes a favorable operating balance of $11,176.09, which is slightly over 2 per cent of current receipts, compared to operating deficits of $27,911.11 in 1946 and $2322.60 in 1947 and a favorable operating balance in 1948 of $61,127.37.

An informative analysis of current receipts and disbursements can be made on the basis of the "income dollar" and how it was used. Thus, of each dollar of current receipts in 1949,

39.4 cents came from dues,
1.0 cent from entrance fees,
50.4 cents from publication sales,
5.5 cents from advertising,
1.8 cents from interest, and
1.9 cents from miscellaneous sources.

We used this income dollar by spending:

38.7 cents for development and publication of standards and reports,
19.9 cents for research, papers and discussions, and
39.2 cents for administration — membership and finances, meetings, sales expense, certain publications, promotional activities, etc.,—leaving
2.2 cents over for surplus account available for Society operations.

The auditors' report shows total assets in all funds (general funds, building fund, and special and designated funds) of $602,778.75, compared to $584,943.65 a year ago, the increase representing principally increased funds held for the technical committees of the Society. The report also includes a summary of the comparative financial position of the Society for the past five years. The surplus in the General Funds account on December 31, 1949, was $219,235.36 compared to $227,160.50 a year ago, a decrease of $7925.14. The ratio of surplus to operating disbursements at current levels dropped from about 50 to 45 per cent.

Building Fund.—The auditors' report also includes a statement of cash receipts and disbursements in the Building Fund as well as a balance sheet for the Fund as of December 31, 1949. The cost of the Society Headquarters Building as of the close of the fiscal year was $185,344.87, towards which there has been received a total of $169,482.17 in contributions from members. Other receipts during the life of the Building Fund are shown in the report. The only indebtedness on the building is a 4 per cent note payable to the General Funds of the Society, originally $17,000 in amount but reduced during the fiscal year to $13,866.67.

The auditors' report also shows that there was paid into the Building Fund from General Funds of the Society during the fiscal year the sum of $5700 as a reserve for depreciation, this constituting a charge in the general budget against headquarters occupancy expense. This added to two previous similar payments since the inception of the Fund of $8700 sets up a depreciation reserve of $14,400, so that Society Headquarters are now carried as a depreciated asset of $170,944.87.

Investments.—Investments of Society funds as of December 31, 1949, are given in the auditors' report, both book and market values being shown. As of that date the book value of all investments was $264,913.38; the market value of these investments was $249,065.96, a depreciation of approximately 6 per cent. On the basis of market values 63.2 per cent were in bonds, 9.6 per cent in preferred stock, and 27.2 per cent in common stocks; Government bonds constituted 55.6 per cent of all investments.

The following changes during the fiscal year in the investment portfolio are noted:

GENERAL FUNDS

Bought	*Cost*
$24,600 U. S. Savings Bonds Series G	$24 600.00
Federal Intermediate Credit Bank Debentures	8 000.00
100 shares Atlantic Refining Co.	3 326.85
100 shares Chase National Bank	3 412.50
50 shares Gulf Oil Corp.	3 098.97
100 shares Ohio Oil Co.	2 623.35
100 shares Philadelphia Electric Co.	2 334.41
	$47 396.08

Called or Matured	*Received*
$2000 Associated Electric Co. Bonds (called)	$2 000.00
Federal Intermediate Credit Bank Debentures (matured)	24 600.00
$20,000 U. S. Treasury Bonds 1949–51 (called)	20 000.00
	$46 600.00

RESEARCH FUND

Bought	*Cost*
$5400 U. S. Savings Bonds Series G	$5 400.00
Federal Intermediate Credit Bank Debentures	2 000.00
	$7 400.00

Called or Matured	*Received*
$2000 Associated Electric Co. Bonds (called)	$2 000.00
Federal Intermediate Credit Bank Debentures (matured)	5 400.00
	$7 400.00

The net profit on the calling of securities in the General Funds as reported above was $31.50; the profit on calling of securities noted above in the Research Fund was $306.50.

No account has been taken in the accompanying financial statement of the assets of the Society in the form of publications in stock. An inventory of the principal technical publications as of May 1, 1950, may be summarized as follows:

Year Book (1949)	564
Proceedings (all back copies not incl. 1949)	2 890
Index to Proceedings	550
1949 Book of A.S.T.M. Standards	
Part 1	5 350
Part 2	4 576
Part 3	[a]
Part 4	4 777
Part 5	3 926
Part 6	[a]
A.S.T.M. Methods of Chemical Analysis	589
Compilations of Standards	18 355
Selected Standards for Students	3 324
Symposiums and Special Reprints	75 917
Reprints of Standards (approximate)	160 000
Index to A.S.T.M. Standards (1948)	408
Radiographic Standards for Steel Castings	36

1950 Finances:

In preparing the budget for 1950, the Board has estimated current receipts at $560,000, made up of $205,000 from dues and entrance fees, $301,000 from publication sales, and $54,000 from miscellaneous sources, principally advertising, income from investments, exhibit, and registration and other fees. The figure for publication sales includes the sum of $115,000 estimated receipts from sales of the current Book of Standards. To current receipts will be added $750 from the Retirement Fund Reserve to be applied towards the increased costs of the Retirement Plan this year, as mentioned in the following section of this report, making total estimated receipts of $560,750.

The budget of disbursements has been fixed at $550,000. As usual, the two principal items are cost of publications at $244,000 and salaries at $186,500, comprising together about 78 per cent of estimated disbursements. The budget for publications provides for all regular and special publications anticipated in 1950, including the sum of $10,000 for printing technical papers presented at the Pacific Area meeting last October in either *Proceedings*, BULLETIN or special publications. There is also included $8000 to complete payment of the costs of the 1949 Book of Standards (estimated at $137,000) after applying the $40,000 publication reserve accumulated during the past two years to this item—the majority of this cost having been met in 1949 financing. Another important item in the publication budget is the sum of $40,000 set aside as a reserve towards publication of the 1952 Book of Standards, with the expectation of setting aside the same amount in 1951. These reserves, considered with the costs of the Supplements to the Book of Standards in 1950 and 1951, will spread the costs of publishing the official standards of the

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[a] Printing not completed as of May 1, 1950.

Society fairly evenly over the three year period 1950–51–52.

The salary budget provides for the staff of 53 as of January 1, 1950, authorized by the Board, with provision for an additional stenographer and certain extra clerical services. The stenographer was obtained in April, 1950, bringing the headquarters staff to 54.

It will be seen that the budget as set up anticipates an operating surplus of $10,750.

A review of operations under this budget during the first five months of 1950 indicates that receipts and disbursements thus far are about as estimated and no change in the budget seems necessary at this time.

To complete reference to 1950 finances thus far this year, it is noted that during the first five months the following maturity, sales and purchases of securities have taken place:

In General Funds: Matured, $8000 Federal Intermediate Credit Bank Debentures; sold, 25 shares American Can Co. Common, 50 shares American Cyanamid Co. Common, 100 shares Atlantic Refining Co. Common, 10 shares American Cyanamid Co. Preferred, and $3000 Southern Pacific bonds; purchased, 25 shares Allis-Chalmers Manufacturing Co. Cumulative Convertible Preferred, 25 shares Philco Corp. Preferred, 50 shares Servel Corp. Cumulative Preferred, 25 shares Allis-Chalmers Manufacturing Co. Common, 75 shares American Tobacco Co. Common, 50 shares Gulf Oil Corp. Common, and 15 shares Montgomery Ward and Co. Common.

In the Research Fund: Matured, $2000 Federal Intermediate Credit Bank Debentures; purchased, 35 shares Montgomery Ward and Co. Common.

In the Building Fund: Purchased, $5000 U. S. Savings Bonds Series G.

In Committee Funds: Purchased, $5000 U. S. Savings Bonds Series G.

The total book value of Society investments as of May 31, 1950, in all funds was $275,302.57. The Society's cash balance in General Funds as of the same date was $269,973.06.

Retirement Plan for Employees

During the year there has been a net increase of two persons participating in the Retirement Plan—three losses (two by resignation, one by death) and five additions—bringing the total number of participants to 30 out of 47 eligible employees.

The initial retirement annuity policies taken out for the five new participants just mentioned are "fully matched" policies, the employees paying premiums of 2½ per cent of salary and the Society matching. Additional policies were taken out in May, 1950, for twelve participants to bring their retirement annuities up to the requirements of the Plan, namely, 1 per cent per year of service up to age 65 for men and 60 for women, times the average of the five highest salary years, or of $5000 if the salary is above this figure. These policies are of the "unmatched" type, the Society contributing either all or nearly all of the required premiums and the employees contributing only enough to keep their total contributions to the Plan up to 2½ per cent of salary or of $5000, whichever is smaller.

For 13 of the 30 participants, including those whose salaries are over $5000, all retirement policies have now been taken out that are required under the Plan.

The Society's total contribution in premium deposits for retirement policies for the above-mentioned 30 employees is at the annual rate of approximately $15,000, which is roughly 8 per cent of the present salary roll and $2\frac{1}{2}$ per cent of the estimated current income. The total of employees' annual contributions, including insurance benefits which most of them have elected, is approximately $3500.

Cooperative Activities

The Society's many cooperative relations with other organizations have continued during the year along lines of well-established policy, and mention will be made here of only a few of the more significant happenings and certain new developments during the year.

Cooperation with the Federal Specifications Board and its various technical committees has been continued to the mutual advantage of Federal and A.S.T.M. specification work. Contacts at the staff level are maintained, which serve to keep the staffs and through them the committees of the two organizations informed of important developments in specification activities. Committees of the Society continue to review many Federal specifications in draft form.

The A.S.T.M. Ordnance Advisory Committee, acting upon a request of the Ordnance Department, has appointed a panel to review proposed military specifications for "Steel: Parts for Ordnance Materiel" now under consideration in the Ordnance Department. Similar panels appointed two years ago to advise on subjects relating to plastics, adhesives, and rubber have continued their activities this year as circumstances required.

The Society has joined with the Technical Association of the Pulp and Paper Industry (TAPPI) in forming a Joint Committee on Petroleum Wax, which, so far as A.S.T.M. is concerned, will function under the procedure of Committee D-2 on Petroleum Products and Lubricants as one of its technical committees. The Joint Committee was organized on February 20, 1950, in New York City, during the annual meeting of TAPPI; A. M. Heald of the Marathon Corp. was elected chairman, B. B. Killingsworth of Socony-Vacuum Oil Corp. vice-chairman, and A. P. Anderson of Shell Oil Co. secretary. Representation of the Society's Committee D-6 on Paper and Paper Products has been arranged. As usual in activities of this kind, the Joint Committee will report independently to the two sponsor societies (in the case of A.S.T.M. through Committee D-2), each organization reserving the right to independent action. The Joint Committee has begun work on a number of methods of test initiated by an earlier TAPPI committee.

To adjust a conflict in scope that had developed with The American Society of Mechanical Engineers in connection with some activities in Committee D-2 on the subject of oil systems of turbines, it has been arranged (1) that Committee D-2 will appoint a representative on the ASME Cooperating Committee on Lubricants; (2) that ASME will be invited to become a member of Committee D-2 with representation on Technical Committee C on Turbine Oils and other groups in which they may be interested; and (3) that Section 1 on Oil Systems of Turbines of Technical Committee C will be set up as a joint ASME-ASTM group. It is understood that the procedures to be worked out in this group will relate primarily to design and operation as distinguished from methods of testing so that primary jurisdiction for publication would lie with ASME; but it has been arranged that A.S.T.M. may if deemed desirable publish certain procedures as information only, as a means of extending publicity to this work.

Arrangements have been made with the American Society for Quality Control for that Society to review the individual sections of the revised and enlarged Manual on Presentation of Data upon which Committee E-11 on Quality Control of Materials is working. If these sections meet with the endorsement of ASQC, that fact will be noted on the respective sections of the Manual when printed.

The Board has accepted an invitation for the Society to be represented on the Research Council of the Graphic Arts Industry where it is felt that A.S.T.M. may be in a position through its work in various technical fields such as paper and printing inks, to contribute helpfully to the technical progress of the graphic arts. The Technical Secretary of the Society has been named as interim representative pending the appointment of an official Society representative.

Following are certain appointments on cooperative activities made during the year:

W. C. Voss has been re-appointed as the Society's representative on the Division of Engineering and Industrial Research of the National Research Council.

F. L. LaQue has succeeded C. D. Hocker as the Society's representative on the Inter-Society Committee on Corrosion of the National Association of Corrosion Engineers. J. S. Pettibone of the A.S.T.M. staff was appointed as an alternate on this committee, succeeding C. S. Cole, deceased.

The Executive Secretary was appointed as a member of the Industry Advisory Committee for Supply Cataloging of the Munitions Board.

Sydney Werthan has been appointed as the Society's representative on the Joint Committee on Paint, Varnish, and Lacquer, succeeding H. A. Nelson, deceased.

I. V. Williams has been appointed as the Society's representative on the Non-Ferrous Metals Division of the Society of Automotive Engineers, succeeding C. S. Cole, deceased.

American Standards Association:

The Society's technical relationships with the work of the American Standards Association come largely under the responsibility of the Administrative Committee on Standards, and the report of that committee (see Appendix II) discusses a number of matters that have developed during the year.

The Board has noted the status of the efforts of the American Standards Association to obtain a federal charter of incorporation. A bill for such incorporation was introduced in the last Congress but died with the adjournment of that Congress, and was introduced in modified form in the Senate of the present Congress where it has been referred to the Senate Committee on Judiciary. The Board of Directors has expressed its opinion to ASA that it believes it is unnecessary for ASA to be federally incorporated and inopportune to take the matter up at this time.

The Board, acting for the Society, approved amendments to the Constitution and By-laws of ASA whereby the title of the chief executive officer of ASA was changed to Managing Director.

The Conference of Executives of Organization Members (CEOM) of ASA has continued its activities which are proving valuable in acquainting the executive officers of member bodies of ASA with the activities of the Association and in stimulating standardization activities in industry. Two meetings of the Conference have been held during the year, and on a third occasion the Conference was invited by the Board of Directors of ASA to meet with it for a discussion of problems and activities of the Association. The Executive Secretary is a member of the Executive Committee of CEOM.

C. H. Rose was appointed for a three-year term expiring December 31, 1952, as Society representative on the ASA Standards Council, succeeding F. M. Farmer who retired from this post after many years of distinguished service on the Council, including a term as its chairman. The other Society representative on the Council is J. R. Townsend, now its vice-chairman; J. H. Foote and the Technical Secretary are alternates on the Council.

The following appointments have been made on four of the Correlating Committees of ASA: G. H. Harnden on Miscellaneous Projects, H. K. Nason on Chemical Industry, M. N. Clair (reappointed) on Building Codes, and M. P. Davis (reappointed) on Safety Codes.

International Activities

The principal contacts of the Society with international activities have been in connection with various standardization projects being undertaken in the International Organization for Standardization (ISO). Brief reference to the status of a number of these projects is contained in the report of the Administrative Committee on Standards (Appendix II) to which reference should be made. The Society continues to maintain contact with a number of the standardizing bodies of other countries through exchange of standards for review and comment, exchange of publications, and in other ways. Cooperation with the British Institution of Petroleum in the development of oil measurement tables has continued and is mentioned more specifically in an earlier section of the report.

Respectfully submitted on behalf of the Board of Directors,

J. G. MORROW,
President.

C. L. WARWICK,
Executive Secretary.
June, 1950.

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR JANUARY 1 TO DECEMBER 31, 1949

Philadelphia, January 6, 1950

Mr. C. L. Warwick, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pennsylvania

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the year ended December 31, 1949. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the records. Accounts receivable were not verified by correspondence with debtors. The investments owned by the Society were examined by us; all income from investments for the period under review was properly accounted for.

We have prepared and submit herewith balance sheet as of December 31, 1949, statement of cash receipts and disbursements for the year ended that date, and other supporting schedules, all of which are in agreement with the records of the Society.

In our opinion, these statements, with notes thereto, present fairly the financial position of the Society at December 31, 1949 and the results of its operations for the year ended that date.

Respectfully submitted,

John Heins & Co.

BALANCE SHEET AS OF DECEMBER 31, 1949

(Including Special and Designated Funds)

ASSETS

General Funds:			
Cash	$153 379.22		
Less Check No. 2266 as of December 16, 1949 (drawn against cost of 1949 Proceedings and 1949 Book of Standards)	97 000.00		
	$56 379.22		
Investments (Market Value $182 016.43)—Cost	192 325.57		
Accounts Receivable	22 516.20		
Note Receivable (Building Fund)	13 866.67		
		$285 087.66	
Furniture and Fixtures (depreciated book value)		18 078.39	
Total Assets General Funds			$303 166.05
Building Fund:			
Cash		$6 739.02	
Investments (Market Value $8 805.47)—Cost		8 632.83	
Land (Cost)		30 000.00	
Building and construction costs	$155 344.87		
Less Reserve for depreciation	14 400.00	140 944.87	
Total Assets Building Fund			$186 316.72
Other Special and Designated Funds:			
Cash: A.S.T.M. Research Fund	6 460.79		
Dudley Medal and Marburg Lecture Fund	1 509.61		
Richard L. Templin Award Fund	58.96		
Sam Tour Award Fund	24.50		
Committee Funds	41 287.14		49 341.00
Investments:			
A.S.T.M. Research Fund (Market Value $42 499.21)—Cost	45 829.98		
Dudley Medal and Marburg Lecture Fund (Market Value $3 995.00)—Cost	6 625.00		
Richard L. Templin Award Fund (Market Value $1 035.94)—Cost	1 000.00		
Sam Tour Award Fund (Market Value $1 443.00)—Cost	1 500.00		
Committee Funds (Market Value $9 270.91)—Cost	9 000.00	63 954.98	
Total Assets Special and Designated Funds			$113 295.98
Total Assets			602 778.75

LIABILITIES

General Funds:			
Accounts Payable:			
Committee C-1	$149.31		
Advance Collections	12 200.46		
Life Membership Fund	3 812.50		
Publication Fund	1 841.80		
Book of Standards Reserve	40 000.00		
Executive Retirement Reserve	11 000.00		
Retirement Fund Reserve	2 296.62		
Reserve for Depreciation of Investments	12 000.00	83 930.69	
Surplus		219 235.36	
Total Liabilities General Funds			$303 166.05
Building Fund:			
Contributions from Members		169 482.17	
Contributions from A.S.T.M. General Funds		1 755.01	
Accumulated Income and Profit on Investments		1 212.87	
Note Payable to A.S.T.M. General Funds		13 866.67	
Total Building Fund			186 316.72
Other Special and Designated Funds:			
A.S.T.M. Research Fund:			
Principal	45 914.60		
Income	6 376.17		
		52 290.77	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 625.00		
Income	1 509.61		
		8 134.61	
Richard L. Templin Award Fund:			
Principal	1 000.00		
Income	58.96		
		1 058.96	
Sam Tour Award Fund:			
Principal	1 500.00		
Income	24.50		
		1 524.50	
Committee Funds, Unexpended Balances		50 287.14	
Total Special and Designated Funds			$113 295.98
Total Liabilities and Funds			$602 778.75

COMPARISON OF GENERAL FUNDS FOR FISCAL YEARS 1945–1949 INCLUSIVE.

At Close of Fiscal Year	Assets				Liabilities			
	Cash	Investments	Accounts Receivable	Furniture and Fixtures	Accounts Payable	Reserve for Book of Standards	Miscellaneous Funds	Surplus
1945	$46 058.60	$153 718.05	$15 033.90	$6 641.57	$5 432.16	$15 000.00	$35 982.49	$165 037.47
1946	5 100.03	160 383.60	26 150.27	14 964.50	7 270.11	15 000.00	40 117.54	144 210.75
1947	3 930.42	163 690.12	37 218.85	16 405.39	7 755.95	20 000.00	36 070.74	157 418.09
1948	50 691.65	191 497.99	46 748.11	17 261.45	7 777.78	40 000.00	31 260.92	227 160.50
1949	56 379.22	192 325.57	36 382.87	18 078.39	12 349.77	40 000.00	31 580.92	219 235.36

RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD JANUARY 1, 1949 TO DECEMBER 31, 1949

Cash balance, January 1, 1949			$50 691.65
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$191 473.41		
Past Dues	1 448.00		
Advance Dues	4 886.25		
Income, Life Membership Fund	175.00		
Entrance Fees	4 799.00		
Total Dues		$202 781.66	
Sales of Publications:			
Book of Standards (Members, addl. parts)	38 390.00		
1946 Book of Standards	29 576.95		
1947 Supplements to Book of Standards	13 168.38		
1948 Supplements to Book of Standards	39 550.79		
1949 Book of Standards	2 456.70		
Methods of Chemical Analysis of Metals	2 039.65		
Compilations of Standards	53 767.25		
Separate Standards	15 003.19		
Selected Standards for Students	11 759.09		
Proceedings	6 194.45		
Spring Meeting Papers	2 259.93		
Bulletin Subscriptions (non-members)	1 945.36		
Special and Miscellaneous Publications	37 015.20		
Total Sales of Publications		$253 126.94	
Miscellaneous:			
Advertising—BULLETIN	25 310.08		
Advertising—Index	2 160.00		
Interest and Dividends	8 849.48		
Registration and Other Fees—Meetings	7 135.00		
Committee A-1 for Technical Assistant	655.00		
ASTM-DCC Reference Fuel Account	843.14		
Miscellaneous	1 186.22		
Total Miscellaneous Items		$46 138.92	
Total Operating Receipts (budgeted)		$502 047.52	
Nonoperating Receipts (Not Budgeted):			
Investments Matured or Sold	46 600.00		
Excess Remittances	720.20		
Committee C-1 for Technical Assistant	2 400.00		
Retirement Fund Reserve	320.00		
Joint AIME-ASTM Publication Fund	142.30		
Institute of Petroleum Publications	194.90		
Joint Comm. on X-ray Diffraction	285.00		
From Building Fund on Acct. of Loan	3 133.33		
From Advisory Comm. on Corrosion, return of loan	5 000.00		
Miscellaneous	350.00		
Total Nonoperating Receipts		$59 145.73	
Total Receipts			$561 193.25
Total Receipts for 1949 and Cash Balance January 1, 1949			$611 884.90

DISBURSEMENTS

Operating Disbursements (Budgeted):			
Publications:			
Year Book	$10 705.96		
1946 Book of Standards	739.43		
1949 Book of Standards	85 216.04[a]		
Compilation of Standards	10 099.66		
Separate Standards	4 967.11		
Chemical Analysis of Metals	279.81		
Proceedings	25 031.99[a]		
Preprints	7 663.89		
Spring Meeting Papers	1 609.40		
A.S.T.M. Bulletin	23 363.22		
Circulars to Members	2 656.79		
Index to Standards	6 557.05		
Special and Miscellaneous Publications	26 189.38		
Total Disbursements—Publications		$205 079.73	
Salaries	172 731.95		
General Office Expenses	42 124.31		
Expenses—Technical Committees	10 691.49		
Expenses—Meetings	17 322.05		
Exhibit, Annual Meeting	277.51		
American Standards Association	1 000.00		
Traveling Expenses, Administrative and Special Committees	4 452.44		
Headquarters Occupancy Expenses (includes $5 700 depreciation on building)	17 694.08		
Furniture and Fixtures	3 404.56		
Dues, Contributions, Miscellaneous	1 394.76		
Legal Services	1 000.00		
Employees Retirement Fund	13 698.55		
		285 791.70	
Total Operating Disbursements (Budgeted)		$490 871.43	
Nonoperating Disbursements (Not Budgeted):			
Refund of Excess Remittances	720.20		
Investments	47 396.08		
Technical Assistant Committee C-1	2 400.00		
Jt. Comm. X-ray Diffraction	9 017.97		
Loan to Advisory Committee on Corrosion	4 000.00		
Miscellaneous	1 100.00		
Total Nonoperating Disbursements		$64 634.25	
Total Disbursements			$555 505.68
Cash Balance, December 31, 1949			$56 379.22

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[a] **These accounts include $97 000.00 representing a check drawn against cost of the 1949 Proceedings and 1949 Book of Standards, but not actually paid on December 31, 1949.**

A.S.T.M. BUILDING FUND

DETAILS OF BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD JANUARY 1, 1949 TO DECEMBER 31, 1949

Cash Balance, January 1, 1949		$578.22
Receipts		
Contributions from Members	$3 075.05	
Contributions from A.S.T.M. General Funds	1 755.01	
Interest on U. S. Treasury Bonds 2½s 1967–72	212.50	
From General Funds for Depreciation on Building, Elevator and Air Conditioning Installation	5 700.00	10 742.56
		$11 320.78
Disbursements		
Air Conditioner	$440.75	
Sound Proofing	386.00	
Interest on Loan from General Funds	621.68	
Reduction of Loan from General Funds	3 133.33	4 581.76
Cash Balance, December 31, 1949		$6 739.02

A.S.T.M. BUILDING FUND

DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF DECEMBER 31, 1949

Assets

Cash in bank				$6 739.02
Investments:				
$8500 U. S. Treasury Bonds 2½s 1967–72 (Market value $8805.47)—Cost				8 632.83
Property, 1916–18 Race Street, Philadelphia, Pa.:				
Purchase Price	$53 625.00			
Commission to Agent	2 681.25			
Settlement Fees, net	197.10			
	$56 503.35			
Allocated to:				
Land			$30 000.00	
Building		$26 503.35		
Construction Costs:				
Architect's Fee	$11 544.70			
Rent paid to facilitate possession	110.00			
Zoning fees and permits	17.50			
General Contractor	86 933.72			
Air Conditioning	20 749.00			
Elevator	7 548.04			
Air Conditioning Units	650.75			
Exhaust system for basement	216.25			
Real Estate Agent's Fee	500.00			
Soundproofing	907.00			
Miscellaneous	817.48			
	$129 994.44			
Less proceeds from sales and salvage of equipment and rentals	1 152.92			
		128 841.52		
		$155 344.87		
Reserve for Depreciation (provided from General Funds)		14 400.00	140 944.87	170 944.87
Total Assets				$186 316.72

Liabilities and Funds

Contributions from Members			$169 482.17	
Contributions from A.S.T.M. General Funds			1 755.01	
Accumulated Income:				
Interest on U. S. Treasury 2½s 1967–72		$1 958.28		
Profit on disposal of securities		76.27		
		2 034.55		
Less Interest on Loan from General Funds		821.68	1 212.87	
Note payable to A.S.T.M. General Funds			13 866.67	$186 316.72

In addition to the General Funds of the Society, the Treasurer had on hand the following funds as of December 31, 1949:

A.S.T.M. RESEARCH FUND

	Total	*Invested*	*Un-invested Cash*
Principal Account:			
Balance, January 1, 1949	$45 608.10	$45 523.48	$84.62
Receipts:			
Sale of Securities		−7 400.00	+7 400.00
Profit on Sale of Securities	306.50	+306.50	
Disbursements:			
Purchase of Securities		+7 400.00	−7 400.00
	$45 914.60	$45 829.98	$84.62
Income Account:			
Balance, January 1, 1949		$5 392.92	
Receipts:			
Interest and Dividends on Investments		1 483.55	6 876.47
Disbursements:			
Welding Research Council		$500.00	
Bank Charge for postage		.30	500.30
Balance, December 31, 1949			$6 376.17

DUDLEY MEDAL AND MARBURG LECTURE FUND

Balance, January 1, 1949			
Principal—Investment (at cost)	$6 625.00		
Income—Cash in bank	1 501.70	$8 126.70	
Receipts			
Interest on Investments		330.00	$8 456.70
Disbursements			
Honorarium		200.00	
Dudley Medals		68.50	
Miscellaneous (Travel exp. etc.)		53.59	322.09
			$8 134.61
Accounted for as follows:			
Principal—Investments (at cost)		$6 625.00	
Income—Cash in bank		1 509.61	$8 134.61

RICHARD L. TEMPLIN AWARD FUND

Balance, January 1, 1949			
Principal—Investments (at cost)	$1 000.00		
Income—Cash in bank	60.46	$1 060.46	
Receipts			
Interest on Investments		25.00	$1 085.46
Disbursements			
Templin Award		25.00	
Engrossing Certificates		1.50	26.50
			$1 058.96
Accounted for as follows:			
Principal—Investments (at cost)		$1 000.00	
Income—Cash in bank		58.96	$1 058.96

SAM TOUR AWARD FUND

Balance, January 1, 1949			
Principal—Investments (at cost)	$1 500.00		
Income—Cash in bank	37.50	$1 537.50	
Receipts			
Interest on Investment	37.50		
Contribution from General Funds	50.00	87.50	$1 625.00
Disbursements			
Tour Award	35.00		
Electro and Certificate	65.50		100.50
			$1 524.50
Accounted for as follows:			
Principal—Investments (at cost)		$1 500.00	
Income—Cash in bank		24.50	$1 524.50

COMMITTEE FUNDS

Committee A-1 on Steel	$450.97	
Committee A-5 on Corrosion of Iron and Steel	1 138.55	
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	2 427.48	
Committee B-6 on Die-Cast Metals and Alloys	2 642.29	
Committee B-8 on Electrodeposited Metallic Coatings	11.25	
Committee C-1 on Cement	783.73	
Committee C-1 Cement Reference Laboratory	900.00	
Committee C-9 Sanford E. Thompson Medal Fund	923.52	
Committee C-9 on Concrete and Concrete Aggregates	153.32	
Committee C-15 on Manufactured Masonry Units	394.28	
Committee C-18 on Natural Building Stones and Slate	176.36	
Committee D-1 on Paint, Varnish, Lacquer and Related Products	546.68	
Committee D-2 on Petroleum Products and Lubricants	43.64	
Committee D-2 D.C.C. Reference Fuel Account	19 699.58	
Committee D-4 on Road and Paving Materials	36.95	
Committee D-5 on Coal and Coke	1 505.24	
Committee D-9 on Electrical Insulating Materials	777.78	
Committee D-12 on Soaps and Other Detergents	233.40	
Committee D-13 on Textile Materials	396.65	
Committee D-13 on Research-Textile Finishes	238.34	
Committee D-15 on Engine Antifreezes	43.00	
Committee E-3 on Chemical Analysis of Metals	144.39	
Committee E-9 on Fatigue	197.56	
ASA Sectional Committee on Specifications for Case Iron Pipe and Special Castings	1 563.04	
Detroit District Council	3 814.10	
Philadelphia District Council	397.68	
Philadelphia District Council (Annual Meeting Acct.)	108.45	
Pittsburgh District Council	55.50	
Western New York-Ontario District Council	217.57	
Advisory Committee on Corrosion	7 491.76	
Administrative Committee on Ultimate Consumer Goods	201.55	
Joint Committee on X-ray Diffraction	381.15	
Joint Committee on X-ray Diffraction Research Associate	2 191.38	
Total Committee Funds		**$50 287.14**
Accounted for as follows:		
Cash in bank and on hand	$41 287.14	
Investments (at cost)	9 000.00	**$50 287.14**

INVESTMENTS

AS OF DECEMBER 31, 1949

GENERAL FUNDS

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
$4 000	Erie R. R. Co. Gen. Mtg. Income 4½s, 2015	$2 832.00	$2 720.00
2 000	Hudson Coal Co. S. F. 5s Series A 1962	1 697.25	1 862.50
3 000	Southern Pacific Deb. 4½s, 1969	2 544.75	2 895.00
37 600	U. S. Savings Bonds, Series G 2½s	37 600.00	36 560.80
4 800	U. S. Treasury Bonds 2½s, 1967/72	4 800.00	4 972.50
2 000	U. S. Treasury Bonds, 2½s, 1964/69	2 000.00	2 065.00
2 000	U. S. Treasury Bonds, 2s, 1952/54	2 000.00	2 031.25
50 000	U. S. Treasury Bonds, 2s, 1952/54	50 765.63	50 781.25
5 000	Walworth Co. 3¼s convertible debentures 1976	5 496.53	4 437.50
8 000	Federal Intermediate Credit Bank Deb.	8 000.00	8 000.00
		$117 736.16	$116 325.80
Shares	*Preferred Stocks*		
22	American Airlines Inc. 3½ cumulative convertible	2 244.00	1 540.00
10	American Cyanamid Co. 3½ Series A convertible	1 042.53	1 200.00
25	Colgate-Palmolive-Peet Co. $3.50	2 525.00	2 493.75
100	Murray Corp. of America Cumulative 4 per cent	5 050.06	3 700.00
20	Philco Corp. 3¾ Series A	2 030.00	1 680.00
		$12 891.59	$10 613.75
	Common Stocks		
75	Allis-Chalmers Mfg. Co., no par value	3 316.20	2 456.25
25	American Can Co.	2 122.30	2 662.50
50	American Cyanamid Co.	1 764.25	2 537.50
100	American Rad. & Std. Sanitary Corp.	1 564.32	1 387.50
25	American Tobacco Co.	2 110.12	1 862.50
30	American Viscose Corp.	2 323.58	2 175.00
100	Atlantic Refining Co.	3 326.85	3 862.50
100	Baldwin Locomotive Works	3 822.00	1 100.00
100	Carpenter Steel Co.	5 426.00	2 975.00
10	Delaware Power and Light Co.	135.00	213.75
75	General Telephone Corp.	2 675.00	2 212.50
50	Gulf Oil Corp.	3 098.97	3 225.00
300	International Harvester	9 610.00	8 325.00
60	Lone Star Cement, no par value	4 349.42	4 560.00
30	Montgomery Ward Co.	1 813.99	1 646.25
100	Ohio Oil Co.	2 623.35	2 750.00
67	Philadelphia Electric Co.	1 185.06	1 633.13
100	Philadelphia Electric Co.	2 334.41	2 437.50
116	Public Service Elec. & Gas Co. avg.	4 369.50	2 972.50
20	United Gas Improvement Co.	315.00	532.50
100	Chase National Bank	3 412.50	3 550.00
	TOTAL Common Stocks	$61 697.82	$55 076.88
	TOTAL Common and Preferred Stocks	$74 589.41	$65 690.63
	TOTAL Stocks and Bonds	$192 325.57	$182 016.43

A.S.T.M. BUILDING FUND

Par Value	*Bonds*		
$8 500	U. S. Treasury, 2½s, 1967–72	$8 632.83	$8 805.47

A.S.T.M. RESEARCH FUND

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*[a]
1 500	B. & O. R. R. Gen. & Ref. 5s Ser. M, 1996	$1 511.25	$900.00
2 000	Hudson Coal Co. S.F. 5s Ser. A, 1962	1 697.25	1 862.50
9 900	U. S. Defense Savings Bonds, Ser. G, 2½s	9 900.00	9 610.20
2 000	U. S. Treasury Bonds 1965/70, 2½s	2 000.00	2 028.12
2 000	Federal Intermediate Credit Corp. due 4.3.50	2 000.00	2 000.00
		$17 108.50	$16 400.82
Shares	*Preferred Stocks*		
30	Allis-Chalmers Mfg. Co. 3¼ cumulative convertible	3 007.22	2 625.00
33	American Airlines Inc. 3½ cumulative convertible	3 366.00	2 310.00
30	Butler Bros., cumulative	3 090.00	2 145.00
12	Consolidated Edison Co. of N. Y. $5	1 234.39	1 305.00
29	Consumers Power Co., $4.50, no par value	2 914.50	3 233.50
20	Philco Corp., 3¾, Series A	2 030.00	1 680.00
		$15 642.11	$13 298.50
	Common Stocks		
100	Allis-Chalmers Mfg. Co., no par value	4 421.59	3 275.00
5	Delaware Power & Light Co.	67.50	106.88
98	General Motors Corp., par $10—gift[b]	6 504.75	7 019.25
20	Montgomery Ward Co.	1 209.33	1 097.50
33	Philadelphia Electric Co.	583.70	804.38
9	Public Service Elec. & Gas Co.	135.00	230.63
10	United Gas Improvement Co.	157.50	266.25
	TOTAL Common Stocks	$13 079.37	$12 799.89
	TOTAL Common and Preferred Stocks	$28 721.48	$26 098.39
	TOTAL Stocks and Bonds	$45 829.98	$42 499.21

DUDLEY MEDAL AND MARBURG LECTURE FUND

Par Value	*Bonds*		
$500	B. & O. Ref. & Gen. Mtg. 6s, Series J, 1995	$550.00	$350.00
6 000	B. & O. R.R. Ref. & Gen. Mtg. 5s, Series G, 1995.	6 075.00	3 645.00
		$6 625.00	$3 995.00

RICHARD L. TEMPLIN AWARD FUND

$1 000	U. S. Treasury Bonds 2½s, 1967–72	$1 000.00	$1 035.94

SAM TOUR AWARD FUND

$1 500	U. S. Defense Savings Bonds, Series G 2½	$1 500.00	$1 443.00

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[a] Market values were taken from current financial publications as of close of market December 31, 1949, except U.S. Savings Bonds Series G, which are stated at redemption value as of December 31, 1949.

[b] The income from these shares is, by agreement, paid to an annuitant.

COMMITTEE FUNDS

Par Value	*Bonds*	*Cost or Book Value*	*Market Value*
$1 000	U. S. Treasury Bonds, 2⅞s, 1955–60	$1 000.00	$1 084.06
2 000	U. S. Treasury Bonds, 2½s, 1964–69	2 000.00	2 065.00
3 000	U. S. Treasury Bonds, 2½s, 1965–70	3 000.00	3 042.15
3 000	U. S. Treasury Bonds, 2½s, 1967–72	3 000.00	3 079.70
		$9 000.00	$9 270.91
	GRAND TOTALS, Bonds	$161 602.49	157 276.94
	GRAND TOTALS, Preferred Stocks	28 533.70	23 912.25
	GRAND TOTALS, Common Stocks	$74 777.19	67 876.77
		$264 913.38	249 065.96

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

In pursuance of its principal function, the Administrative Committee on Standards has acted upon numerous tentatives and revisions which have been submitted since the Society's Annual Meeting in June, 1949. Hereinafter is included a complete record of such recommendations as have been approved.[1] These actions include 43 new tentatives accepted during the year as well as revisions of 65 tentatives, tentative revisions of 13 standards, 10 standards reverted to tentative, and 5 standards withdrawn. A number of these were acted upon at a meeting of the committee held on September 22, 1949, and the remainder were handled by correspondence.

The committee has continued its efforts toward improvement of quality and form of standards. At the instance of the committee, the Board of Directors has arranged with Committee E-1 for preparation of a manual on the form of A.S.T.M. methods for guidance of the technical committees. It is planned that when this manual is completed it will be put in the hands of subcommittee and group leaders engaged in the writing of methods of testing as well as in the hands of technical committee chairmen and officers. It is hoped that this procedure will insure more adequate treatment of certain important features such as editorial form, accuracy, reproducibility, etc.

The Administrative Committee is also in the process of adding to its regulations a procedure which has already been established to advise those having a negative viewpoint of their privilege of being present at meetings of the Administrative Committee on Standards at which recommendations that concern them are being considered. This is one additional step to insure proper consideration of all negative viewpoints prior to acceptance of technical committee recommendations.

During the year the committee has also studied several projects looking to the establishment of new technical committees in the Society. Some of these investigations have resulted in setting up new committees and others are still in process of organization.

Expansion of Standardization Activities

Of the projects referred to in the report of a year ago, organization has now been effected of the following committees:

C-22 on Porcelain Enamel, and

D-21 on Wax Polishes and Related Materials.

The subject of graphite, referred to in the previous report, has been referred to Committee D-2 on Petroleum Products and Lubricants with the thought that that committee would undertake work on graphite when used as a lubricant, leaving for future development the question as to whether any work need be undertaken with respect to other uses of graphite.

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[1] This list is not reproduced here, since the recommendations have all been announced currently throughout the year in the ASTM Bulletin.—Ed.

Committee C-3 on Chemical-Resistant Mortars.—Some years ago the question of work on sulfur cements was explored, and it was the consensus that a new committee on the subject should be established. More recently Committee C-12 on Mortars for Unit Masonry established a subcommittee on acid-resistant mortars, but this work together with the personnel of the subcommittee expanded to such an extent that on recommendation of Committee C-12 the Board of Directors authorized the establishment of work in this field as a separate project and accordingly Committee C-3 on Chemical Resistant Mortars has been organized.

Smooth Surface Floor Coverings.—Mention was made in the report a year ago that it had been proposed to organize a new committee to deal with asphalt floor tile and other smooth surface floor coverings. Accordingly a canvass was conducted and subsequently a conference was held at Society Headquarters to examine into the desirability of organizing such a committee. The study is being continued.

Other Pending Proposals.—Recently it has been proposed that the Society organize a committee on the use of radioactive isotopes in testing and also a committee dealing with air pollution, having in mind methods of sampling and analysis. It is understood that these proposals are receiving the attention of the Board of Directors.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Association are used by A.S.T.M. in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing A.S.T.M. standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the A.S.T.M. is granted proprietary sponsorship so far as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations but with A.S.T.M. as sponsor or co-sponsor for the sectional committee.

Standards Submitted Under the Existing Standards Procedure.—No new standards were submitted during the past year under the existing standards procedure.

Revised Standards Submitted Under Proprietary Procedure.—In view of the Society having been designated as proprietary sponsor for the revisions of A.S.T.M. standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 1 specification relating to slab zinc, 11 relating to copper and copper alloys, 3 relating to structural shapes, 1 relating to paint pigment, 2 relating to linseed oil, and 2 relaing to detergents.

Standards Submitted Under Sectional Committee Procedure.—During the year reports were received from five of the sectional committees for which the Society is sponsor or joint sponsor as follows:

Sectional Committee A1 on Specifications and Methods of Test for Hydraulic Cements.—This committee has recommended the approval as American Standard of 3 specifications, the approval of 9 revised American Standards, and the reaffirmation of 4 American Standards.

Sectional Committee B36 on Wrought-Iron and Wrought-Steel Pipe and Tubing.—This committee has recommended the approval as American Standard of 6 specifications and the approval of 6 revised American Standards.

Sectional Committee J6 on Rubber Protective Equipment for Electrical Workers.—This committee has recommended the approval as American Standard of 4 specifications.

Sectional Committee L14 on Textile Test Methods.—This committee has recommended the approval of 5 revised American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 6 methods, approval of 9 revised American Standards, and the reaffirmation of 4 American Standards.

All of these reports have been referred to the American Standards Association. Action is still pending so far as the recommendations of Sectional Committee A1 are concerned; the others have been approved.

INTERNATIONAL RELATIONS

The Society continues to receive from other countries standards with the request for comment. A.S.T.M. committees review these standards and frequently suggestions result. Apart from such informal cooperation, however, quite a number of proposals have been received through the American Standards Association concerning projects that have been proposed under the International Organization for Standardization (ISO).

Textiles.—The general secretariat for ISO Committee 38 has been assigned to Great Britain, but the United States will hold the secretariat for several subcommittees. A special subcommittee has been set up under Sectional Committee L14 on Textile Test Methods to serve as the American committee in charge of contacts with the ISO Committee.

Petroleum Products.—Sectional Committee Z11 on Petroleum Products and Lubricants is still communicating with American industry to learn its views with respect to Sectional Committee Z11 acting as American Secretariat for this ISO Committee (No. 28), including an expression of willingness or otherwise of the industry to support such a secretariat financially.

Rubber.—The secretariat for ISO Committee 45 is held by Great Britain. Two meetings of the committee have been held, one in London, June, 1948, at which America was represented by Mr. H. G. Bimmerman, member of A.S.T.M. Committee D-11 on Rubber and Rubber-like Materials, and the second at The Hague, Netherlands, at which America was represented by Mr. Etienne de Meeus, also member of Committee D-11. The committee has accepted the invitation of America to hold a meeting in Akron during October, 1950.

Viscosity.—The matter of the desirability of the United States participating in this international project was referred to Committee E-1 on Methods of Testing, which committee felt that international discussion on the subject would be desirable provided it dealt with absolute viscosity.

Plastics.—Committee D-20 on Plastics is at the present time considering the desirability of recommending that America participate in this ISO project, and further, the desirability of America's accepting the secretariat.

Shellac.—Subcommittee XIII on Shellac of A.S.T.M. Committee D-1 has been set up as the American committee in charge of contacts with the ISO Committee on Shellac (No. 50) for which

India holds the secretariat, and has submitted a number of comments to the ISO Committee with respect to standards dealing with shellac that are now under consideration. A meeting of the ISO committee was held in Delhi, India, January, 1950.

Solid Mineral Fuels.—Committee D-5 on Coal and Coke is being looked to to handle American contacts with this ISO Committee No. 27. A meeting of the ISO committee was held in London during March, 1950.

Iron and Steel.—Committee A-1 on Steel has indicated that it favors American participation in the work of this ISO Committee No. 17 on the basis of an observer.

Chemistry.—The following A.S.T.M. committees have indicated an interest in participating in the ISO Project on Chemistry:

D-1 on Paint, Varnish, Lacquer, and Related Products,
D-3 on Gaseous Fuels, and
D-12 on Soaps and Other Detergents.

Laboratory Glassware.—An ISO Project on Laboratory Glassware (No. 48) has been proposed with the British Standards Institution holding the secretariat. Two conferences of representatives of industry in this country have been held at which the Society was represented. These conferences have recommended that the scope of the international project be clarified and delimited before any further consideration is given to American participation.

General Definitions Relating to Chemical and Physical Test Results.—Committee E-11 on Quality Control has indicated that it favors American participation in the work of this ISO committee (No. 69).

Mica.—Subcommittee IX on Mica of A.S.T.M. Committee D-9 has been set up as the American committee in charge of contacts with the ISO Committee on Mica (No. 56) for which India holds the secretariat, and has submitted a number of comments to the ISO Committee with respect to standards dealing with mica that are now under consideration. A meeting of the ISO Committee was held in Delhi, India, in January at which America was represented by Messrs. U. C. Lal and Peter DeCicco, representatives in India of the Asheville-Schoonmaker Mica Co.

Copper and Copper Alloys.—Committee B-5 on Copper and Copper Alloys has concurred in the recommendation of the Copper and Brass Research Association that America participate in the work of this ISO Committee (No. 26). The secretariat for this committee is held by Great Britain.

Respectfully submitted on behalf of the committee,

R. D. BONNEY,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

The Administrative Committee on Research has not met since presenting its annual report to the Board of Directors a year ago. Routine matters requiring action by the committee were handled by correspondence to conserve time and expense, such as the decision to continue the annual contribution of $250 to the Welding Research Council in support of researches in the welding field that are of value to the Society.

In its report last year, the committee cited the discussions it has carried on with the technical committees of the Society regarding committee organization for research and the listing of unsolved testing problems in the various fields of work in A.S.T.M. With respect to the latter question, considerable interest has been expressed by the technical committees, several of which have now submitted formal statements of research problems patterned upon a form of presentation suggested by the Administrative Committee. Other committees are engaged in the development of such statements. It had been hoped that by March or April there would have been enough such statements in hand to warrant a meeting of the Administrative Committee for consideration, but it was decided to give the technical committees more time and to accumulate a sizeable number of such statements before holding a meeting. Accordingly, the Administrative Committee can only report that progress is being made on this project.

It is gratifying to be able to report that staff contacts with the technical committees have revealed several instances where extensive researches are being considered and plans developed for raising substantial sums of money to support such researches. Two such projects now being discussed with the appropriate committee are studies to determine the effect of moisture on thermal conductivity of a variety of insulating materials and a study of the "tunnel test" for determining fire resistances of various types of finishes. The Joint Committee on Chemical Analysis by X-ray Diffraction Methods is engaged in raising funds to continue the research fellowship established last year at the National Bureau of Standards, and the Advisory Committee on Corrosion has raised over a third of the $100,000 fund being sought to support the A.S.T.M. Exposure Test Site program. Another extensive research project for the testing of wood poles is being developed by Committee D-7 on Wood, for which funds of the order of $100,000 will be sought. The 1950 annual reports of the technical committees will doubtless contain many references to interesting research activities in various stages of planning and development.

The Society has continued its ties with the Division of Engineering and Industrial Research of N.R.C., and with the Highway Research Board and the Building Research Advisory Board of that Division.

Respectfully submitted on behalf of the committee,

WALTER C. VOSS,
Chairman.

C. L. WARWICK,
Secretary.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The activities of the Administrative Committee on Papers and Publications have been largely those of reviewing material for publication and in planning for its publication, and also in planning the technical program for the Annual Meeting. Accordingly, this report is largely a record of the various regular and special publications that have been issued during the past year. The amount of technical papers being handled by the Society has been greatly increased this year due to the Pacific Area National Meeting held in San Francisco last October. As a result of the meeting, six special technical publications have resulted and a number of other papers are being published in the ASTM BULLETIN and others will appear in the 1950 *Proceedings*.

A special Study Committee was set up a year ago to review the Society's standards for A.S.T.M. papers with the purpose of raising the standards if possible. This committee, consisting of G. R. Gohn, chairman, R. C. Adams, and K. B. Woods, developed a modified detailed check chart for the use of reviewers of technical papers. Such a chart has been in use for a year and has proved to be very helpful both to the reviewer as well as to the Headquarters Staff. The committee also prepared a brochure containing suggestions to authors with respect to preparation and presentation of technical papers. It is hoped that this will be available within the next few months. An article entitled "ASTM Papers—Their Preparation, Acceptance, and Publication" was published by the committee in the January issue of the BULLETIN, and the Manual on Preparation of Technical Papers (but without the appended material) was published in the April issue of the BULLETIN. It is felt that when the complete Manual becomes available it will be of considerable assistance to authors in the preparation of technical papers. Quite a bit of interest has already been expressed in it by individuals, companies and schools.

It will be recalled that last year the number of issues of the ASTM BULLETIN was increased from six to seven. This year eight issues are scheduled as previously planned.

RECORD OF PUBLICATIONS ISSUED THIS YEAR

Regular Publications:

1949 Proceedings, 1252 pp., 7000 copies
1949 Book of Standards
  Part 1, 1402 pp., 12,750 copies
  Part 2, 1160 pp., 11,000 copies
  Part 3, 1372 pp., 10,000 copies. Available May, 1950
  Part 4, 1321 pp., 10,000 copies
  Part 5, 1730 pp., 9,600 copies
  Part 6, 1410 pp., 9,000 copies. Available May, 1950
1949 Index to A.S.T.M. Standards, 280 pp., 20,000 copies. Available in May, 1950.
ASTM BULLETIN, 8 issues (May, 1949, to April, 1950), total number of pages 728, average number of copies 9800.

A statement of the volume of the principal publications for the past four years is given below:

Publications	1946 pages	1947 pages	1948 pages	1949 pages
Proceedings	1614	1206	1366	1252
Book of A.S.T.M. Standards	6694	....	....	8395
Supplements to Book of A.S.T.M. Standards	....	2015	1648	....
A.S.T.M. Methods of Chemical Analysis of Metals	412	....	....	....
Index to A.S.T.M. Standards	240	254	272	280
Year Book	512	542	569	570
Spring Meeting Symposiums	166	119	....	....
ASTM BULLETIN	600	668	664	728
	10238	4804	4519	11225

Special Compilations of Standards Published from April 15, 1949, to April 15, 1950:

A.S.T.M. Specifications for Steel Piping Materials (A-1), 328 pp., 2500 copies.

A.S.T.M. Specifications for Flat Steel Products (A-1, A-10), 200 pp., 1000 copies. Available May, 1950.

A.S.T.M. Standards on Copper and Copper Alloys (B-5), 508 pp., 1200 copies.

A.S.T.M. Specifications and Tests for Electrodeposited Metallic Coatings (B-8), 61 pp., 7000 copies.

A.S.T.M. Standards on Cement (C-1), 224 pp., 2400 copies. Available July, 1950.

A.S.T.M. Standards on Mineral Aggregates, Concrete, and Nonbituminous Highway Materials (C-9, D-4), 248 pp., 2400 copies. Available May, 1950.

A.S.T.M. Standards on Glass and Glass Products (C-14), 120 pp., 1000 copies. Available May, 1950.

A.S.T.M. Standards on Paint, Varnish, Lacquer and Related Products (D-1), 687 pp., 2000 copies.

A.S.T.M. Standards on Petroleum Products and Lubricants (D-2), 752 pp., 7600 copies.

A.S.T.M. Standards on Gaseous Fuels (D-3), 98 pp., 750 copies.

A.S.T.M. Standards on Coal and Coke (D-5), 166 pp., 1800 copies.

A.S.T.M. Standards on Electrical Insulating Materials (D-9), 678 pp., 1200 copies.

A.S.T.M. Standards on Rubber and Rubber-Like Materials (D-11), 744 pp., 1800 copies. Available June, 1950.

A.S.T.M. Standards on Soaps and Other Detergents (D-12), 178 pp., 1500 copies.

A.S.T.M. Standards on Textile Materials (D-13), 588 pp., 2800 copies.

A.S.T.M. Standards on Adhesives (D-14), 62 pp., 1500 copies.

A.S.T.M. Standards on Industrial Aromatic Hydrocarbons (D-16), 44 pp., 750 copies.

Procedures for Testing Soils, including ASTM Standards (D-18), 425 pp., 5000 copies. Available August, 1950.

A.S.T.M. Standards on Industrial Waters (D-19), 142 pp., 1000 copies.

A.S.T.M. Standards on Plastics (D-20), 570 pp., 2200 copies. Available June, 1950.

1950 Appendix to ASTM Manual of Engine Test Methods for Rating Fuels, 32 pp., 2250 copies.

Report of Joint Committee on Standard Specifications for Concrete and Reinforced Concrete, 146 pp., 1200 copies.

Special Publications Also Issued:

1949 Edgar Marburg Lecture.—"Residual Stresses in Metals" by William March Baldwin, Jr., 49 pp., 2000 copies.

Symposium on Speed of Testing, 87 pp., 2000 copies (reprinted from 1948 Proceedings).

A.S.T.M. Manual on Presentation of Data, STP 15-C, 83 pp., 1000 copies.

X-Ray Diffraction Data for Chemical Analysis, Second Supplement, STP 48-B, 1337 cards, 1000 sets.

Revised Original set and 1st Supplement, 2723 cards, 1000 sets.

1948 Supplement to the Bibliography and Abstracts on Electrical Contacts, STP 56-E, 24 pp., 1200 copies.

Symposium on Effects of Low Temperature on Materials, STP 78, 68 pp., 4000 copies.

Symposium on Magnetic Testing, STP 85, 204 pp., 3000 copies.

Symposium on Metallography in Color, STP 86, 90 pp., 3000 copies.

Symposium on Deformation of Metals as Related to Forming and Service, STP 87, 123 pp., 2000 copies.

Symposium on Industrial Gear Lubricants, STP 88, 23 pp., 2000 copies.

Symposium on Aging of Rubbers, STP 89, 78 pp., 4000 copies.

Metal Cleaning Bibliographical Abstracts, STP 90, 72 pp., 2000 copies.

Manual on Fatigue Testing, STP 91, 86 pp., 4000 copies.

Symposium on Lubrication of High-Speed Turbine Gear Equipment, STP 92, 32 pp., 2000 copies.

Symposium on Accelerated Durability Testing of Bituminous Materials, STP 94, 187 pp., 1800 copies.

Symposium on Insulating Oils (Third Series), STP 95, 59 pp., 2000 copies.
Symposium on Testing Cast Iron with SR-4 Type of Gage STP 97, 96 pp., 1500 copies.

Symposiums Reprinted from ASTM BULLETIN:

Round Table Discussion on the Need for Standards for the Inspection of Water-Borne Industrial Wastes, 12 pp., 500 copies.

Reprints of Publications:

Symposium on Insulating Oils—Series 1 and 2, 48 pp., 500 copies.
Hardenability Chart, 20,000 copies.
Viscosity Temperature Charts:
  Chart B, 21,000 copies
  Chart C, 10,000 copies
  Chart E, 11,600 copies
Knock Test Manual Framing Test Chart, 15,000 copies.
Standard Specifications for Portland Cement Stucco and Portland Cement Plastering, 20 pp., 2000 copies.

Special Publications in Prospect:

Data on Compositions and Properties of Corrosion and Heat-Resistant Steels and Alloys—Wrought and Cast, STP 52-A, 84 pp., 2000 copies.
1949 Supplement to the Bibliography and Abstracts on Electrical Contacts, STP 56-F, 24 pp., 1200 copies.
Monograph on Papers and Paper Products, STP 60-A, 110 pp., 2000 copies.
Symposium on Evaluation Tests for Stainless Steels, STP 93, 246 pp., 3000 copies.
Symposium on Radiography, STP 96, 64 pp., 2000 copies.
Symposium on Rapid Methods for the Identification of Metals, STP 98, 120 pp., 2000 copies.
Symposium on Use of Pozzolanic Materials in Mortars and Concretes, STP 99, 240 pp., 3000 copies.[1]
The Strength of Wrought Steels at Elevated Temperatures, by R. F. Miller and J. J. Heger, STP 100, 114 pp., 1600 copies.
Papers on Ultrasonic Testing, STP 101, 75 pp., 2000 copies.
Symposium on High Additive Content Oils, STP 102, 70 pp., 3000 copies.[1]
Papers on Application of Statistics, STP 103, 31 pp., 2500 copies.[1]
Papers on Dynamic Stress Determinations, STP 104, 75 pp., 3000 copies.[1]
Symposium on Turbine Oils, STP 105, 60 pp., 2000 copies.[1]
Papers on Bituminous Paving Mixtures, STP 106, 64 pp., 2000 copies.[1]
A.S.T.M.-I. P. Manual of Oil Measurement Tables (STP number not yet assigned), 1100 pp., 3000 copies.

CURRENT ACTIVITIES

A full program is in prospect for the 1950 Annual Meeting with a number of symposiums scheduled as follows:

Symposium on Effect of Sigma Phase on the High Temperature Properties of Metals—under the auspices of the Joint Committee on Effect of Temperature on the Properties of Metals.
Symposium on Corrosion of Gas Turbine Materials—under the auspices of the Joint Committee on Effect of Temperature on the Properties of Metals.
Symposium on Methods of Testing Soils Under Triaxial Loading—under the auspices of Committee D-18 on Soils for Engineering Purposes.
Symposium on Identification and Classification of Soils—under the auspices of Committee D-18 on Soils for Engineering Purposes.
Symposium on the Role of Non-Destructive Testing in the Economics of Production—under the auspices of Committee E-7 on Non-Destructive Testing.

In addition to the above, there are a number of other technical papers which will be presented at the Annual Meeting. A number of papers are being offered to the Society for publication but not presentation at a meeting. These for the most part will be published currently in the ASTM BULLETIN.

Of the special publications listed as being in prospect, the one covering the volume correction tables, "A.S.T.M.-I.P. Manual of Oil Measurement Tables," is of greatest magnitude. This represents a monumental undertaking on the part of Committee D-2 on Petroleum Products in the preparation of these tables, and their reproduction represents quite a publication problem.

As indicated earlier, a considerable amount of the publication program consists of the papers presented at the

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[1] Comprising paper presented at the Pacific Area National Meeting in October, 1949.

Pacific Area National Meeting held in San Francisco. Some of these papers have been appearing in current issues of the ASTM BULLETIN, and others will appear in the 1950 *Proceedings.* For the most part, however, it is planned to publish these as special technical publications, some of which are listed above as being in immediate prospect. In accordance with an understanding at the time of the meeting, it is not intended to undertake the publication of certain of the Pacific Area meeting papers.

Respectfully submitted on behalf of the committee,

C. L. WARWICK,
Chairman.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON ULTIMATE CONSUMER GOODS

The Administrative Committee on Ultimate Consumer Goods held a meeting on May 17, 1950, at the offices of the United States Testing Co., Hoboken, N. J.

In the last report reference was made to certain changes in the Regulations Governing Technical Committees relating to a procedure for setting up standards for ultimate consumer goods which had been formulated by the Administrative Committee, tentatively approved by the Board of Directors, and, towards the close of last year, was submitted to the chairmen of all technical committees for criticism and comment. Since that time the Administrative Committee has considered, at the request of the Board, certain proposals from one technical committee for an alternative wording. After due consideration the Administrative Committee reaffirmed its original proposal and so reported to the Board with its reasons therefore. The Committee understands that the Board has since formally approved the proposed revisions in the Regulations and that these are now in effect.

The Advisory Group on Consumer Want Determination has been requested by the Quartermaster Corps to review its plans for determining the wantability of certain fabrics which have been prepared for officers' summer uniforms. Through Paul S. Olmstead, Chairman of the Advisory Group, the Administrative Committee has been advised of the proposed plan whereby the preference of the officers is to be determined, and of the criticisms and suggestions which were made by the Advisory Group for improvement of the technique. It is expected that the experience gained from this study of want determination may be especially illuminating because of the greater control over variables which is possible with this selected class of individuals than is possible in the case of studies which involve groups of the general public.

The Administrative Committee has approved tentative plans for a Symposium on the Determination of Wantability of Consumer Goods to be held at the annual meeting of the Society in 1951. The papers will present both the theoretical and practical approaches to the problem. The plans include giving extensive publicity to the symposium to members of the Society and to other interested groups.

The Administrative Committee has formulated a program which will bring to the attention of the technical committees of the Society some of the earlier literature which the Committee distributed, and the procedure which has been made available to committees that wish to do work in the consumer goods field. A paper is also planned for the October issue of the ASTM Bulletin which will discuss phases of the field of work assigned to the Administrative Committee and their pertinence to the aims and objectives of the Society. It is intended to use reprints of the paper to inform organizations interested in consumer goods of what the Society has done to permit its technical committees to work in the consumer goods field.

The Administrative Committee has considered the work of ASA Sectional Committee on Rayon Fabrics, L-22, and has advised the Board of Directors of certain features which should be noted.

The Administrative Committee calls attention to an important change in the organization of Committee D-13 on Textiles. The two subcommittees which dealt most closely with ultimate consumer goods, namely, the Subcommittee on Household and Garment Fabrics and the Subcommittee on Hosiery, have been merged into one under the title of Subcommittee on Ultimate Consumer Textile Products. This subcommittee will place its emphasis on test methods for consumer goods.

Respectfully submitted on behalf of the committee,

HERBERT J. BALL,
Chairman.

R. E. HESS,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

The Administrative Committee on Simulated Service Testing has not held a meeting during the current year due, primarily, to the change in status of its 1949–50 program. Committee plans, made at the last meeting, were premised upon the presentation this year of a symposium on Bearings and Lubricants, as well as a reconsideration of the back log of proposals submitted to A.C.S.S.T. Further survey of the proposed symposium revealed the desirability of postponing this presentation to 1951, because of the greater potentialities for up-to-date developments now in process. Also, the untimely death of our sterling Secretary, Carter Cole, necessitated a full survey of committee activities and proposals before calling another meeting.

A re-survey of all proposals before the A.C.S.S.T. is under way and a meeting of the Administrative Committee is being held during the present Annual Meeting for the purpose of planning the 1950–51 agenda of activities.

The tentative agenda for the committee consideration include:

1. Bearing Symposium,
2. Strength of Soldered Joints,
3. Gasket Materials,
4. Airborne Dirt, etc.,
5. Fracture Tests (as re: welded structures, etc.),
6. Low-Temperature Properties of Metals.

In addition to the above, a number of the committee back-log items such as adherence of coatings, hardness, fungus growth, flexure testing, hydraulic packing, strain gages, etc., are reported to be receiving some attention in one or more technical committees. These items will be considered from the standpoint of their applicability in all phases of Society work.

It is proposed that this meeting of A.C.S.S.T. have in attendance the members of the Technical Staff at Headquarters; it being considered that through these men who are actively in contact with all A.S.T.M. technical committees that A.C.S.S.T. can have available the latest specific information as to Society status, activities, etc., in the various subjects brought before the committee.

There are no recommendations to the Board of Directors at this time.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

APPENDIX VII

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

Since there have been no matters of an administrative or policy nature requiring the attention of the Administrative Committee on District Activities during the past year, this report essentially refers to certain activities of the A.S.T.M. Districts.

The Administrative Committee increased its personnel last year from six to eight to provide somewhat broader representation. The policy of having a printed return report form was put into effect and has greatly improved the style and continuity of the Annual Report from the Districts.

The committee reports the formal organization of the Ohio Valley District, centered in the Louisville-Cincinnati-Indianapolis-Dayton-Columbus area. This group is planning several meetings and expects to act as the host committee during the 1951 A.S.T.M. Committee Week in Cincinnati in March.

Reference should be made to an extensive questionnaire of the District activities circulated by the New England District Council. A wealth of information was developed concerning meeting matters, organization of the District, distribution of Council minutes, and such matters which will merit further study. The New England group should be commended on its initiative in gathering the concensus of the members' reactions on many questions.

Perhaps acting as a local committee on arrangements or as host to the Society during national meetings might be noted as one of the very important services which the Districts, through their Councils, render. The Northern and Southern California District Councils served as the basis for the General Committee on Arrangements for the Society's First Pacific Area National Meeting, which was an outstanding success. For the 1949 Annual Meeting, the Philadelphia District Council served as the host group and is repeating again for the 1950 Annual Meeting in Atlantic City. During 1950 Committee Week in Pittsburgh, the District Council there arranged an informal cocktail party with the President as the honored guest. The Administrative Committee wishes to commend our Districts and their Councils for contributions of this kind. With such concentration as there is in the Society on technical matters, the Districts, by fostering certain social and entertainment functions during national meetings, help foster a better *esprit de corps*.

Concerning District meetings, one desirable trend has been marked, namely, to enlist the cooperation of the respective local sections and chapters of other organizations in sponsoring these, or at least to invite the membership of the local chapters of other groups to join with A.S.T.M. This policy has developed many friendly relations with other local groups, and it is expected to be continued. With the membership of the Society concerned with such diverse materials and subjects, a large attendance of the membership at District meetings cannot be expected as a rule, and so the excellent attendance at many of the meetings is attributable to the joint sponsorship with other groups.

The Administrative Committee has noted with much interest a recent visit by the Assistant Secretary to a number of western industrial centers, where luncheon or dinner meetings of our members were held, these functions being the first get-togethers of the A.S.T.M. members in these areas. In each instance, the members present felt it would be desirable for some officer of the Society and Staff representative, if feasible the President and Executive Secretary, to visit these areas and our members. While the Administrative Committee does not contemplate any recommendations concerning a more formal organization of our members in areas or cities not now represented in the Districts, it may well be that in the interest of promoting the activities of the Society, some recognition should be taken of areas, particularly in the West and Southwest, where there is continually expanding industrial activity.

Respectfully submitted on behalf of the committee,

FRANK G. STEINEBACH,
Chairman.

R. J. PAINTER,
Secretary.

REPORT OF COMMITTEE A-1

ON

STEEL[1]

During the past year there have been several developments in Committee A-1 of general interest to the Society membership, including general requirement specifications and mechanical testing procedures for classes of products and also discussion on the words "free" and "without charge" included in the Society's general inspection clause.

Early in 1949, as noted in the 1949 Report of Committee A-1, a new arrangement for presentation of its specifications on structural steel was completed. In this new form the structural specifications for specific products include only those requirements which are applicable to the particular product. For requirements generally applicable to structural steel, the individual specifications refer to a general requirement specification titled General Requirements for Delivery of Rolled Steel Plates, Shapes and Bars for Structural Use (A 6 - 49 T).

Committee A-1 is now balloting upon a like arrangement for its boiler plate specifications which has been developed by Subcommittee XI on Steel for Boilers and Other Pressure Vessels. If a favorable letter ballot vote results, the revised individual boiler plate specifications and the proposed Tentative Specifications for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (to be assigned the designation A 20) will be presented to the Administrative Committee on Standards for Society approval subsequent to the Annual Meeting. Subcommittee XV on Bar Steels is considering revision of its specifications in this same manner.

For some time it has been evident to many of the subcommittees that it would be necessary to develop mechanical testing procedures for the field of products under their jurisdiction. Mechanical testing has become so specialized that no one cover-all method could possibly do justice to the details necessary for testing all the various steel products. Consequently the subcommittees have been developing mechanical testing procedures applicable to their products, including bars, pipe and tubing, sheet and strip, etc. The first of these procedures to be letter balloted upon by Committee A-1 is appended to this report as proposed Tentative Methods and Definitions for Mechanical Testing of Steel Bars.[2] Committee A-1 hopes to have the Society publish a manual covering the mechanical testing of steel products when all these various procedures have been approved.

Committee A-1 has had under consideration for over a year the significance of the words "free" and "without charge" which are presently included in the inspection clauses of all steel specifications except those on steel sheet and strip. A special group of Committee A-1 members has been appointed by the chairman to meet with the Executive Secretary of the Society, since broad policy questions are involved.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] These methods were accepted by the Society and appear in the 1950 Supplement to ASTM Book of Standards, Part 1.

Personnel.—Committee A-1 now consists of 271 voting members, including 130 producers, 105 consumers, and 36 general interest members.

Following the resignation of J. J. Laudig of the Delaware Lackawanna and Western Railroad as chairman of Subcommittee VI, A. O. Schaefer of the Midvale Co. has been appointed as chairman and G. E. Baumgardner of the Norfolk and Western Railway Co. as vice-chairman.

A. W. MacLaren of the Carnegie-Illinois Steel Corp. has been appointed as secretary of Subcommittee XV on Bar Steels.

Honorary Membership.—In recognition of their long-time and active services in behalf of Committee A-1, Messrs. H. B. Oatley, W. M. Barr and L. B. Jones, were elected to honorary membership. Messrs. Barr and Jones were active in many subcommittees concerned with materials of interest to the railroads and Mr. Oatley has been especially active in the work involving pipe and tubing and materials for high-temperature service. All three men aided the committee in numerous administrative capacities including service on the Advisory Committee. Mr. Barr has largely relinquished his technical committee work, but Messrs. Jones and Oatley are continuing actively; Mr. Oatley serving as consumer vice-chairman of the main committee, as vice-chairman of Subcommittee IX on Steel Pipe and Tubing, and maintaining other subcommittee contacts as is Mr. Jones. Both men have a wealth of experience which will be available to the committee. Mr. Oatley, as chairman of the A.S.M.E. Boiler Code Committee, has been a most effective liaison between this group and Committee A-1.

Losses by Death.—In June, 1949, the committee lost by death, Edward R. Young (died June 9, 1949), Metallurgical Engineer, Climax Molybdenum Co. of Chicago. He had been particularly concerned with the committee's work on steel castings.

In the death of Oliver C. Cromwell (died March 30, 1950), formerly with the Baltimore and Ohio Railroad and for many years retired, the committee loses one of its oldest members. For many years Mr. Cromwell was active in the committee, contributing greatly from his wide store of knowledge of the needs of the railroad industry.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On December 21, 1949, the Administrative Committee on Standards accepted the following recommendations submitted by Committee A-1:

New Tentative Specifications for:

Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Washers (A 325 – 49 T), and

Alloy-Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 49 T).

Revision of Tentative Specifications for:

Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 – 49 T),

High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 212 – 49 T),

Boiler and Firebox Steel for Locomotives (A 30 – 49 T),

Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (A 285 – 49 T),

Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 48 T),

High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 – 47 T),

Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 – 49 T),

Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 – 49 T),
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 – 49 T),
Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 – 49T),
Manganese - Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 – 47 T),
Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 49 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 48 T),
Steel Machine Bolts and Nuts and Tap Bolts (A 307 – 47 T),
Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 – 48 T), and
Hot-Rolled Carbon-Steel Bars (A 107 – 49 T).

Tentative Revision of Standard Specifications for:

Factory-Made Wrought Carbon-Steel and Carbon - Molybdenum - Steel Welding Fittings (A 234 – 44),
Billet-Steel Bars for Concrete Reinforcement (A 15 – 39),
Rail-Steel Bars for Concrete Reinforcement (A 16 – 35),
Axle-Steel Bars for Concrete Reinforcement (A 160 – 39), and
Heat-Treated Steel Elliptical Springs (A 147 – 39).

Withdrawal of Standard Specifications for:

Steel Helical Springs (A 61 – 39), and
Steel Elliptical Springs (A 62 – 39).

The new and revised tentatives, and the tentative revisions of standards appear in the 1949 Book of A.S.T.M. Standards, Part 1.

On December 22, 1949, the Administrative Committee on Standards accepted the following recommendations:

Revision of Tentative Specifications for:

Carbon-Steel Forgings for Rings for Main Reduction Gears (A 290 – 46 T),
Carbon-Steel and Alloy-Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 – 47 T),
Alloy-Steel Forgings for Nonmagnetic Coil Retaining Rings for Turbine Generators (A 289 – 47 T),
Carbon-Steel and Alloy-Steel Forgings for Pinions for Main Reduction Gears (A 291 – 47 T),
Carbon-Steel and Alloy-Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 47 T),
Carbon-Steel and Alloy-Steel Forgings for Turbine Rotors and Shafts (A 293 – 47 T), and
Carbon-Steel and Alloy-Steel Forgings for Turbine Bucket Wheels (A 294 – 47 T).

Revision and Reversion to Tentative of Standard Specifications for:

Carbon-Steel Forgings for General Industrial Use (A 235 – 46),
Carbon-Steel Forgings for Locomotives and Cars (A 236 – 46),
Alloy-Steel Forgings for General Industrial Use (A 237 – 46),
Alloy-Steel Forgings for Locomotives and Cars (A 238 – 46),
Carbon-Steel and Alloy-Steel Ring and Disk Forgings (A 243 – 46), and
Carbon-Steel Seamless Drum Forgings (A 266 – 47).

The new and revised tentatives appear in the 1949 Book of A.S.T.M. Standards, Part 1.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending for publication as tentative two new specifications and one new method of testing, as appended to this report. Revisions in eighteen tentatives and two standards are recommended as itemized in the Appendix. Editorial revisions in eleven specifications are recommended.

These actions have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Structural Steel for Bridges, Buildings and Rolling Stock:

A joint subgroup with Subcommittee XI appointed to make recommendations for changing the present sliding scale requirements for yield point and elongation in the structural and boiler plate specifications of Committee A-1 has proposed some changes in Subcommittee II specifications, as included in the recommended revisions in the Appendix.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Now being studied by the subcommittee is a proposed document covering mechanical testing procedures and definitions for structural steel products. The opinion has been expressed by Subcommittee II that the bend tests for structural products are useful as a guide to the toughness and ductility of the steel and should be retained in its specifications.

The subgroup on flange *versus* web testing of wide flange beams is assembling data on this subject from Lehigh University, the University of Illinois, Modjeski & Masters, Bethlehem Steel Co., and Carnegie-Illinois Steel Corp.

A proposed Tentative Specification for Steel Sheet Piling, developed and approved by Subcommittee II and appended hereto[4], is being recommended by Committee A-1 for publication by the Society. To accommodate this proposed specification, changes are also being recommended in Specification A 6. Various other changes are recommended for Specification A 6 to bring it abreast of present commercial practice.

Changes are recommended in Tentative Specifications A 7, A 94, A 113, A 131, A 141, A 195, A 242, A 283, and A 284, as outlined in the Appendix.

Subcommittee IV on Spring Steel and Steel Springs:

The Section on Hot-Formed Springs has been attempting to develop a specification for spring steel bars of A.I.S.I. 8600 material, but considerable disagreement has been encountered regarding the carbon content range and the size ranges.

During the past year this subcommittee developed a tentative revision of the Standard Specification for Heat-Treated Steel Elliptical Springs (A 147 – 39) in the form of the Tentative Specifications for Heat-Treated Steel Leaf Springs, accepted by the Administrative Committee on Standards, as described previously in this report.

The Standard Specifications for Steel Helical Springs (A 61) and for Steel Elliptical Springs (A 62) were recommended to be discontinued and this action also has received the approval of the Administrative Committee on Standards.

Subcommittee V on Steel Reinforcement Bars:

At the June, 1949, meeting of Subcommittee V, various tentative revisions to Standard Specifications A 15, A 16, and A 160 for Billet Steel, Rail-Steel and Axle-Steel Bars for Concrete Reinforcement were recommended. These recommendations, after letter ballot approval of Committee A-1, were approved by the Administrative Committee on Standards, as explained previously in this report.

Subcommittee VI on Steel Forgings and Billets:

Revisions in thirteen forging specifications under the jurisdiction of this subcommittee have been approved by the Administrative Committee on Standards, as mentioned previously in this report.

A Subgroup on Macroetch Testing has collected a number of photomacrographs illustrating various common defects encountered in etched cross-sections of steel forgings. It is expected that this group will continue to collect photomacrographs until a set can be assembled to illustrate each of the defects listed in the Tentative Method of Macroetch Testing and Inspection of Steel Forgings (A 317 – 48 T).

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[4] This specification was accepted by the Society and appears in the 1950 Supplement to ASTM Book of Standards, Part 1.

The subcommittee has also appointed a special group to prepare methods for the ultrasonic testing of steel forgings.

Changes are recommended in the Tentative Specifications for Carbon and Alloy Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288), for Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292), for Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293), and for Carbon and Alloy Steel Forgings for Turbine Bucket Wheels (A 294), as outlined in the Appendix.

Subcommittee VII on Rolled Steel Wheels and Steel Tires:

Subcommittee VII has developed a proposed Tentative Specification for Heat-Treated Steel Tires, as appended to this report.[5] Committee A-1 is recommending that this specification be approved and published by the Society.

The subcommittee also recommends that the Standard Specifications for Steel Tires (A 26) be reaffirmed in its present form.

Subcommittee VIII on Steel Castings:

A proposed specification for steel castings for steam turbine casings is being developed in Subcommittee VIII and is expected to be acted upon by Committee A-1 and the Society during 1950.

Various changes in the Tentative Specifications for Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 27), including raising the maximum permissible silicon content, and in the Tentative Specifications for High-Strength Steel Castings for Structural Purposes (A 148), including changing the yield requirement of grade 120–100, are under consideration.

This subcommittee has recommended publication by the Society of a paper by H. A. Schwartz and W. K. Bock, entitled "Reproducibility of Results of Tension Tests on Specimens Prepared from Cast Steel Coupons." This reports work carried out as a research project of the Steel Founder's Society.[6]

Subcommittee IX on Steel Tubing and Pipe:

Presently under consideration by this subcommittee is a proposed tentative specification for tubes for low-temperature applications and a proposed mechanical testing manual for steel tubing and pipe.

Also recommended by Subcommittee IX is an editorial revision in Specifications A 178, A 214, A 226, A 249, and A 250 for Electric-Resistance Welded Tubes, changing the section on the reverse flattening test to read that the specimen be split 90 deg. from each side of the weld, instead of the present 180 deg. Other editorial changes are recommended in Specifications A 53, A 83, A 161, A 179, A 199 and A 200, as outlined in the Appendix.

Two subgroups have been appointed to work in conjunction with similar subgroups appointed by Subcommittee XXII. One subgroup will study and make recommendations in respect to changes in chemical requirements of all specifications for steel tubular products, and the other subgroup will study and clarify the descriptions for heat-treated lots.

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[5] This specification was accepted by the Society and appears in the 1950 Supplement to ASTM Book of Standards, Part 1.

[6] See p. 1167.

Subcommittee XI on Steel for Boilers and Other Pressure Vessels:

The Administrative Committee on Standards has accepted revisions during the past year in Tentative Specifications A 30, A 201, A 203, A 204, A 212, A 225, A 285, A 299, A 301 and A 302, as explained previously in this report.

During the past year Subcommittee XI has completed the rearrangement of its specifications, including the new proposed Tentative Specification for General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Quality, as explained previously in this report.

Also under consideration are proposed specifications for 4 to 6 per cent chromium steel; 1 per cent chromium, $\frac{1}{2}$ per cent molybdenum steel; and $2\frac{1}{4}$ per cent chromium, 1 per cent molybdenum steel.

Subcommittee XIII on Methods of Test:

The title of this subcommittee has been changed from "Methods of Physical Test" to "Methods of Test" to provide coverage for test procedures which may not be physical in nature.

After reviewing the proposed Tentative Methods and Definitions for Mechanical Testing of Steel Bars submitted by Subcommittee XV on Bar Steels, this subcommittee recommends approval of the document, as appended.[2]

Subcommittee XV on Bar Steels:

Subcommittee XV has approved and forwarded to Subcommittee XIII the proposed Tentative Methods and Definitions for Mechanical Testing of Steel Bars.

The subcommittee is recommending changes in the Tentative Specifications for Hot-Rolled Carbon-Steel Bars (A 107) and for Cold-Finished Carbon-Steel Bars and Shafting (A 108) as covered in the Appendix to this report.

Now out to letter ballot of Committee A-1 are revisions in the Tentative Specifications for Medium-Carbon Steel Bars Subject to Mechanical Property Requirements (A 306) and editorial rearrangements of the Tentative Specifications for Heat-Treated Alloy-Steel Bars (A 286) for Alloy-Steel Rounds Suitable for Oil Quenching to End-Quench Hardenability Requirements (A 304), and for Hot-Rolled Alloy-Steel Bars (A 322). It is expected to submit these to the Administrative Committee on Standards subsequent to the Annual Meeting.

A proposed tentative specification for cold-finished alloy-steel bars is now being prepared by the subcommittee.

Subcommittee XIX on Sheet Steel and Steel Sheets:

Under review by the subcommittee are Tentative Specifications A 245 and A 246 for Structural Quality Carbon-Steel Sheets and the Tentative Specifications for Cold-Rolled Strip Steel (A 109). The review of Specifications A 245 and A 246 includes width and overweight tolerances of mill edge sheets. In Specifications A 109, the flatness tolerances are under discussion.

Subcommittee XXII on Valves, Fittings, Piping and Flanges for High-Temperature and Subatmospheric Temperatures:

Accepted by the Administrative Committee on Standards on December 21, 1949, was a revision of Tentative Specifications A 217 covering the ferritic grades of steels at present in Specifications A 217 and A 157. It is expected to develop specifications for the austenitic grades so that the old Tentative Specifications A 217 and A 157, covering both ferritic and austentic grades, may be superseded by one specification covering ferritic and another specification covering austenitic grades.

The subcommittee is recommending that the tentative revision of Standard Specifications A 234 be adopted as standard. This tentative revision covers a 1 per cent chromium, $\frac{1}{2}$ per cent molybdenum grade of material to be used in welding fittings.

Subcommittee XXII is also recommending that grades B4, B11, B12, B13, and B15 be deleted from the Tentative Specifications for Alloy-Steel Bolting Material for High-Temperature Service (A 193), as outlined in the Appendix. Also being considered in the subcommittee are some changes in the Tentative Specifications for Alloy-Steel Bolting for Low-Temperature Service (A 320).

Proposed tentative specifications for seamless and welded steel pipe for low-temperature service are being developed in Subcommittee XXII and will soon be in final draft form for letter ballot vote.

The chairman of Subcommittee XXII has received a proposal on the possibility of formulating specifications for centrifugally cast steel pipe, both carbon and alloy steel. It has developed that Committee A-10 has received a similar request to cover high alloy grades and therefore a joint group has been appointed of Subcommittee XXII and Committee A-10 members.

Subcommittee XXVI on Bolting:

The title of this subcommittee has been changed from "common bolting" to "bolting" since its specification work had expanded beyond the scope of its old title.

It is recommending revisions in the Tentative Specifications for Steel Machine Bolts and Nuts and Tap Bolts (A 307) as outlined in the Appendix.

Accepted by the Administrative Committee on Standards on December 21, 1949, were the Tentative Specifications for Quenched-and-Tempered Steel Bolts and Studs with Suitable Nuts and Plain Washers (A 325). The subcommittee is now recommending further revisions in these specifications which may be referred to the Administrative Committee later this year.

Special Subcommittee on Stainless Tubing Specifications:

Various revisions are being considered for the Tentative Specifications for Seamless and Welded Austenitic Seamless Steel Pipe (A 312) to make the specifications more suitable for Boiler Code work.

A revision is recommended in the Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270), as outlined in the Appendix. The revision is also subject to favorable letter ballot of Committee A-10 before adoption.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, N. L. Mochel.
Vice-Chairman, H. B. Oatley.
Vice-Chairman, T. G. Stitt.
Secretary, J. S. Worth.

This report has been submitted to letter ballot of the committee, which consists of 271 members; 195 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

N. L. MOCHEL,
Chairman.

H. C. LARSON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 - 50 T), and
Cold-Finished Alloy-Steel Bars (A 331 - 50 T).

Revision of Tentative Specifications for:

Boiler and Firebox Steel for Locomotives (A 30 - 49 T),
Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 49 T),
Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 49 T),
Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 49 T),
High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 6 in. and Under in Thickness) (A 212 - 49 T),
Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 - 49 T),
Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (Plates 2 in. and Under in Thickness) (A 285 - 49 T),
High Tensile Strength Carbon-Manganese-Silicon Steel Plates for Boilers and Other Pressure Vessels (A 299 - 49 T),
Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 - 47 T),
Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 - 49 T),
Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 - 49 T),
Heat-Treated Alloy-Steel Bars (A 286 - 46 T),
Carbon-Steel Bars Subject to Mechanical Property Requirements (A 306 - 47 T), and
Hot-Rolled Alloy-Steel Bars (A 322 - 49 T).

Revision and Reversion to Tentative of Standard Specifications for:

Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 - 47), and
Open-Hearth Iron Plates of Flange Quality (A 129 - 47).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 1.

On December 14, 1950, the following recommendations were accepted by the Administrative Committee on Standards:

Revision and Reversion to Tentative of Standard Specifications for:

Billet-Steel Bars for Concrete Reinforcement (A 15 - 39),
Rail-Steel Bars for Concrete Reinforcement (A 16 - 35),
Axle-Steel Bars for Concrete Reinforcement (A 160 - 39), and
Minimum Requirements for the Deformation of Deformed Steel Bars for Concrete Reinforcement (A 305 - 49).

The new tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 1, and are published as a group in a special pamphlet.

The following recommendations submitted by Committee A-1 were accepted by the Administrative Committee on Standards on January 25, 1951:

New Tentative Specifications for:

Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 - 50 T).

Tentative Revision of Standard Specifications for:

Carbon- and Alloy-Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 - 48),
Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings (A 234 - 50), and
Heat-Treated Carbon-Steel Bolting Material (A 261 - 47).

Revision of Tentative Specifications for:

Alloy-Steel Bolting Materials for High-Temperature Service (A 193 - 49 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 - 49 T),
Structural Steel for Ships (A 131 - 50 T),
Mild-to-Medium-Strength Carbon-Steel Castings for General Application (A 27 - 46 T),
High-Strength Steel Castings for Structural Purposes (A 148 - 46 T),
General Requirements for Delivery of Rolled Steel Plates of Flange and Firebox Qualities (A 20 - 50 T),
Open-Hearth Iron Plates of Flange Quality (A 129 - 50 T),

- **Carbon-Silicon Steel Plates of Intermediate Tensile Ranges for Fusion-Welded Boilers and Other Pressure Vessels (A 201 - 49 T),**
- High Tensile Strength Carbon-Silicon Steel Plates for Boilers and Other Pressure Vessels (Plates 6 in. and Under in Thickness) (A 212 - 49 T),
- Boiler and Firebox Steel for Locomotives (A 30 - 49 T),
- Chromium-Manganese-Silicon (CMS) Alloy-Steel Plates for Boilers and Other Pressure Vessels (A 202 - 50 T),
- Low and Intermediate Tensile Strength Carbon-Steel Plates of Flange and Firebox Qualities (Plates 2 in. and Under in Thickness) (A 285 - 49 T),
- Nickel-Steel Plates for Boilers and Other Pressure Vessels (A 203 - 49 T),
- Molybdenum-Steel Plates for Boilers and Other Pressure Vessels (A 204 - 49 T),
- Manganese-Vanadium Steel Plates for Boilers and Other Pressure Vessels (A 225 - 49 T),
- Chromium-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 301 - 49 T),
- Manganese-Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 302 - 49 T),
- Seamless Alloy-Steel Pipe for High-Temperature Service (A 158 - 49 T).

Revision and Reversion to Tentative of Standard Specifications for:

- Alloy-Steel Castings for Valves, Flanges, and Fittings for High-Temperature Service (A 157 - 44),
- Seamless Alloy-Steel Boiler and Superheater Tubes (A 213 - 46), and
- Heat-Treated Steel Helical Springs (A 125 - 39).

The new and revised tentatives together with the tentative revisions of the standards appear in the 1950 Supplement to Book of ASTM Standards, Part 1.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various steel products. These specifications appear in their present form in the 1949 Book of A.S.T.M. Standards, Part 1.

NEW TENTATIVES

The committee recommends the following three new tentatives for publication as tentative:

Tentative Specification for Steel Sheet Piling:

This specification is intended to fill the need for a purchase specification for carbon steel sheet piling of structural quality for use in the construction of dock walls, sea walls, cofferdams, excavations and like applications. It is recommended that this specification, as appended hereto,[1] be accepted for publication as tentative.

Tentative Specification for Heat-Treated Steel Tires:

Four classes of heat-treated carbon-steel tires are covered by this specification as follows:

Class A, driving tires for locomotives in passenger service.

Class B, driving tires for freight locomotives and tires for trailer wheels.

Class C, driving tires for switching locomotives, and tires for locomotive trucks, tender trucks, trailer and car wheels.

Class D, driving tires for switching locomotives and heavily loaded trailer tires without brakes.

It is recommended that this specification, as appended hereto,[1] be accepted for publication as tentative.

Tentative Methods and Definitions for Mechanical Testing of Steel Bars:

These methods and definitions have been developed to fill the need for a single document covering the various mechanical testing methods applicable to steel bars. It is recommended that these methods and definitions, as appended hereto,[1] be accepted for publication as tentative.

REVISIONS OF TENTATIVES

The committee recommends revisions in the following eighteen tentatives:

Tentative Specification for General Requirements for Delivery of Rolled Steel Plates, Shapes, and Bars for Structural Use (A 6 - 49 T):

Title.—Add the words "Sheet Piling" after the word "Shapes."

Section 1.—Add the words "sheet piling" after the word "shapes", and in the list of specifications add a reference to the new Tentative Specification for Steel Sheet Piling.

Section 2.—Add a new Paragraph (*c*) to read as follows, relettering the present Paragraph (*c*) as Paragraph (*d*): "(*c*) *Sheet Piling.*—Steel sheet piling consists of rolled sections which can be interlocked, so that a continuous wall is formed when individual pieces are driven side by side."

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[1] These new tentatives were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 1.

Section 5.—Add a new Paragraph (*c*) to read as follows, relettering the present Paragraph (*c*) as Paragraph (*d*): "(*c*) *Sheet Piling.*—The weight of each steel sheet pile shall not vary more than 2½ per cent from the theoretical or specified weight. The length of each steel sheet pile shall not vary more than 5 in. over, and shall not be less than the length specified."

Section 8(c).—Add the words "sheet piling" after the words "structural size shapes" in two places. Change the permissible depth of grinding for material ⅜ in. and over in thickness from the present "1/16" to read "3/32" in.

Add a new Item (*3*) to read as follows, renumbering subsequent items accordingly: "(*3*) The interlock of any sheet piling section may be conditioned by welding and grinding to correct or build up the interlock at any location not to exceed 2 per cent of the total surface area or length of that piece."

Section 9 (*b*).—Revise to read "(*b*) *Shapes.*—Structural shapes and sheet piling shall be marked with the heat number, manufacturer's name, brand, or trade mark, size of section, length, and mill identification marks on each piece; except that small structural shapes with the greatest cross-sectional dimension not greater than 5 in. may be bundled for shipment with each lift marked or tagged showing the above listed identification."

Tables XI and XII.—Combine in one table with changes as shown in the accompanying Table I, renumbering subsequent tables accordingly.

TABLE I.—PERMISSIBLE VARIATION FROM FLATNESS FOR RECTANGULAR SHEARED MILL, UNIVERSAL MILL, CIRCULAR AND SKETCH PLATES.

Specified Thickness, in.	Specified Weight, lb. per sq. ft.	Variation from a Flat Surface for Widths, Lengths or Diameters, Given, in.								
		To 36, excl.	36 to 48, excl.	48 to 60, excl.	60 to 72, excl.	72 to 84, excl.	84 to 96, excl.	96 to 108, excl.	108 to 120, excl.	120 to 144, excl.
To 1/4, excl.	To 10.2, excl.	7/16	5/8	7/8	1	1 1/8	1 1/4	1 1/2	1 5/8	1 3/4
1/4 to 3/8, excl.	10.2 to 15.3, excl.	3/8	9/16	3/4	7/8	1	1 1/8	1 1/4	1 3/8	1 5/8
3/8 to 1/2, excl.	15.3 to 20.4, excl.	5/16	1/2	9/16	11/16	3/4	15/16	1	1 1/8	1 1/4
1/2 to 3/4, excl.	20.4 to 30.6, excl.	1/4	7/16	1/2	5/8	11/16	3/4	15/16	1	1 1/8
3/4 to 1, excl.	30.6 to 40.8, excl.	1/4	3/8	7/16	1/2	5/8	11/16	3/4	13/16	15/16
1 to 2, excl.	40.8 to 81.6, excl.	1/4	5/16	3/8	7/16	1/2	9/16	5/8	11/16	3/4
2 to 4, excl.	81.6	3/16	1/4	5/16	3/8	7/16	1/2	9/16	5/8	11/16
4 to 6, excl.	....	1/4	5/16	3/8	1/2	9/16	5/8	3/4	7/8	1
6 to 8, excl.	....	5/16	7/16	1/2	5/8	3/4	7/8	15/16	1	1
8 to 10, excl.	....	3/8	1/2	5/8	3/4	7/8	1	1	1	1
10 to 12, excl.	....	1/2	11/16	7/8	1	1	1	1	1	1
12 to 15, incl.	....	5/8	13/16	7/8	1	1	1	1	1	1

NOTE 1.—The shorter dimension specified shall be considered the width and the permissible variation from flatness across the width shall not exceed the tabular amount for that dimension.

NOTE 2.—The longer dimension specified shall be considered the length and the permissible variation from flatness along the length shall not exceed the tabular amount for that dimension. When the width or length exceeds 144 in. the variations shown for 144 in. apply for any 12 ft. 0 in. of the specified width or length.

NOTE 3.—The variations given in the above table apply to plates which have a specified maximum tensile strength of not over 72,000 psi., or equivalent hardness. For plates specified to higher tensile strength or hardness, the variations given in the table are customarily doubled.

NOTE 4.—The above table and notes also cover the permissible variations for flatness of circular and sketch plates, based on the maximum dimensions.

Table XIX.—Renumber as Table XVIII and revise the permissible variations in thickness for material under ¼ in. in thickness as follows:

Width, in.	Variations from Thickness, Over and Under, in. Present	Proposed
1 and under	0.006	0.007
Over 1 to 2, incl.	0.008	0.007
Over 2 to 4, incl.	0.010	0.008
Over 4 to 6, incl.	0.010	0.009

Table XX.—Renumber as Table XIX and change the last line of the first column from the present "Over $6\frac{1}{2}$ to 8 in., incl." to read "Over $6\frac{1}{2}$ to $8\frac{1}{4}$ in., incl."

Table XXV.—Renumber as Table XXIV and revise the heading of the fourth column from the present "10 ft. and under" to read "5 to 10 ft. excl." In the headings for the fifth, sixth, seventh, and eighth columns change the word "incl." to read "excl."

Tentative Specification for Steel for Bridges and Buildings (A 7-49T):

Section 7 (a).—Change the present sliding scale figure for minimum elongation in 8 in. of $\frac{\text{“ 1 500 000 ”}}{\text{ten. str.}}$ to "21" per cent.

Tentative Specification for Structural Silicon Steel (A 94 – 49 T):

Section 2.—Revise Paragraph (*b*) to read "In the conditioning of surface imperfections no welding shall be employed except as provided in Paragraph (*c*)."

Add a new Paragraph (*c*) to read "(*c*) If agreed between the manufacturer and the purchaser, welding may be used to condition surface imperfections. In such case, the welding shall be done either with a welding electrode conforming to classification E 6015 or E 6016 of the Tentative Specifications for Mild Steel Arc-Welding Electrodes (A.S.T.M. Designation: A 233),[2] or the area to be welded shall be preheated to a temperature of not less than 212 F."

Section 6.—In Paragraph (*a*) change the present sliding scale values for minimum elongation in 8 in. and 2 in. from the present $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ and $\frac{\text{“1 600 000”}}{\text{ten. str.}}$ to read "16" and "17" per cent, respectively.

In Paragraph (*c*) change the present value for deduction from specified elongation of "0.25 per cent for each $\frac{1}{16}$ in." to "0.125 per cent for each $\frac{1}{32}$ in."

New Section.—Add a new Section 9 titled "Marking" to read "In addition to the standard marking as provided in Specification A 6, material shall be marked with a color identification stripe on one end of each piece."

Tentative Specifications for Hot-Rolled Carbon-Steel Bars (A 107 – 49 T):

Table I.—For all open-hearth free-cutting grades of steel change the maximum permissible phosphorus content from the present "0.045" to read "0.040" per cent.

Tentative Specifications for Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 49 T):

Table I.—Revise as indicated above for Table I in Specifications A 107.

Tentative Specification for Structural Steel for Locomotives and Cars (A 113 – 49a T):

Table I.—Change the present sliding scale figure for minimum yield point of "0.5 tens. str." to read as follows:

Grade A	33,000 psi., min.
Grade B	27,000 psi., min.
Grade C	26,000 psi., min.

Change the present sliding scale figure for minimum elongation in 8 in. of $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ to read as follows:

Grade A	21 per cent, min.
Grade B	24 per cent, min.
Grade C	26 per cent, min.

Change the present minimum elongation in 2 in. for grade B from the present "22" to read "26" per cent.

Reletter the present Footnote *a* as

Tentative Specification for Structural Steel for Ships (A 131 – 49 T):

Table I.—Change the present sliding scale figure for minimum elongation in

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[2] 1949 Book of A.S.T.M. Standards, Part 1.

8 in. of $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ to read “21” per cent for structural steel and “23” per cent for rivet steel.

Change the minimum elongation in 2 in. for structural steel from the present “22” to “24” per cent.

Add a Footnote *a*, referenced after the tensile strength requirements for structural steel to read “The upper limit of 72,000 psi. may be increased by 3000 psi. for material over $1\frac{1}{2}$ in. in thickness.” Reletter the present Footnote *a* as Footnote *b*.

Tentative Specification for Structural Rivet Steel (A 141 – 49 T):

Section 5 (*a*).—Change the present sliding scale figure for minimum elongation in 8 in. of $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ to read “24” per cent.

Tentative Specifications for Alloy-Steel Bolting for High-Temperature Service (A 193 – 49 T):

Section 1.—In Paragraph (*b*) revise the first sentence to read “Eleven grades are covered, including nine ferritic steels and two austenitic steels, designated BA, B5, B6, etc.”

In Note 2 under Paragraph (*d*) in the first sentence change the word “sixteen” to read “eleven.”

Tables I and II.—Delete alloys B4, B11, B12, B13 and B15 from these tables.

Tentative Specification for High-Strength Structural Rivet Steel (A 195 – 49a T):

Section 6 (*a*).—Change the present sliding scale figure for minimum elongation in 8 in. of $\frac{\text{“1 600 000”}}{\text{ten. str.}}$ to read “20” per cent.

Tentative Specification for Low-Alloy Structural Steel (A 242 – 49 T):

Table I.—Change the present sliding scale figure for minimum elongation in 8 in. of $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ to read as follows:

Thickness, in.	Elongation in 8 in., min., per cent
$\frac{3}{16}$ to $\frac{3}{4}$, incl.	18
Over $\frac{3}{4}$ to $1\frac{1}{2}$, incl.	19
Over $1\frac{1}{2}$ to 2, incl.	20

Also for material over $1\frac{1}{2}$ to 2 in., incl., in thickness change the present sliding scale figure of $\frac{\text{“1 600 000”}}{\text{ten. str.}}$ for minimum elongation in 2 in. to read “22” per cent.

Section 6 (*c*).—Revise as indicated above for Section 6 (*b*) in Specification A 94.

Tentative Specification for Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality (Plates 2 in. and Under in Thickness) (A 283 – 49 T):

Table I.—Change the present sliding scale figure for minimum elongation in 8 in. of $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ to read as follows:

Grade A	27 per cent, min.
Grade B	25 per cent, min.
Grade C	23 per cent, min.
Grade D	21 per cent, min.

Reletter the present Footnotes *a* and *b* as Footnotes *b* and *c*, respectively, and add a new Footnote *a*, referenced after the tensile strength requirement for grade D, to read “The upper limit of 72,000 psi. may be increased by 3,000 psi. for material over $1\frac{1}{2}$ in. in thickness.”

Tentative Specification for Low and Intermediate Tensile Strength Carbon-Silicon Steel Plates for Machine Parts and General Construction (A 284 - 49 T):

Section 4.—Revise to read as follows:

4. (*a*) Plates over 4 in. in thickness shall be treated to produce grain refinement either by normalizing or heating uniformly for hot forming. If the required treatment is to be obtained in conjunction with the hot forming operation, the temperature to which the plates are heated for hot forming shall be equivalent to and shall not significantly exceed the normalizing temperature. If the treatment of the plates is not specified to be done at the plate manufacturer's plant, the testing shall be carried out in accordance with Paragraph (*b*).

(*b*) When a fabricator is equipped and elects to perform the required normalizing or fabricates by hot forming as provided in Paragraph (*a*), the plates shall be accepted on the basis of mill tests made on specimens heat treated in accordance with the purchaser's order requirements. If the heat treatment temperatures are not indicated on the purchase order, the plate manufacturer's shall heat treat the specimens under conditions he considers appropriate for grain refinement, and to meet the test requirements. The plate manufacturer's shall inform the fabricator of the procedure followed in treating the specimens at the mill.

(*c*) The purchaser shall indicate in his orders to the plate manufacturer whether the rolling mill or the fabricator shall perform the treatment of the plates.

(*d*) When the plates are to be normalized at the plate manufacturer's plant, the mechanical properties shall be determined on specimens simultaneously treated with the plates.

Table II.—Change the present sliding scale figures of $\frac{\text{“1 500 000”}}{\text{ten. str.}}$ and $\frac{\text{“1 600 000”}}{\text{ten. str.}}$ for minimum elongation in 8 in. and 2 in., respectively, to read as follows:

	Elongation in 8 in., min., per cent	Elongation in 2 in., min., per cent
Grade A............	25	27
Grade B............	23	24
Grade C............	21	22

Tentative Specification for Carbon and Alloy Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 - 49 T):

Table III.—Revise the heading of the third column from the present "Yield Strength (0.01 per cent offset), min., psi." to read "Yield Strength (0.02 per cent offset), min., psi."

Tentative Specification for Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292 - 49 T):

Table III.—Revise as indicated above for Table III in Specifications A 288.

Section 11 (*a*).—Delete the note under this paragraph.

Section 12 (*c*).—Delete the entire paragraph.

Tentative Specification for Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 - 49 T):

Table III.—Revise Footnote *a* to read "At 0.2 per cent offset for class 1 and 0.02 per cent offset for classes 2 to 6, inclusive."

Tentative Specification for Carbon and Alloy Steel Forgings for Turbine Bucket Wheels (A 294 - 49 T):

Table III.—Revise as indicated above for Table III in Specifications A 288.

Section 15 (*a*).—Revise the first sentence of Item (*1*) to read "Forgings up to 30 in., inclusive, in diameter may be group tested."

Tentative Specifications for Steel Machine Bolts and Nuts and Tap Bolts (A 307 - 49 T):

Table I.—Revise the heading of the third column from the present "Mean Thread Area, sq. in." to read "Stress Area, sq. in."

Add the following sentence to Footnote *c*: "No maximum specified for grade A."

Section 12.—Revise Paragraph (*a*) to read "Threads shall be the coarse thread series as specified in the latest issue of the American Standard for Screw Threads (A.S.A. No.: Bl. 1), having. . . ."

Revise the first sentence of Paragraph (*b*) to read "Bolt heads shall be unfinished, regular square, or regular hexagonal, or heavy hexagonal in shape as required, and dimensions shall conform to the latest issue of the American Standard for Wrench Head Bolts and Nuts and Wrench Openings (A.S.A. No.: B 18.2)."

In Paragraphs (*c*) and (*d*) delete the year following the A.S.A. designation and insert the words "the latest issue of," as done above for Paragraphs (*a*) and (*b*).

ADOPTION OF TENTATIVE REVISION AS STANDARD

Standard Specifications for Factory-Made Wrought Carbon-Steel and Carbon-Molybdenum-Steel Welding Fittings (A 234 – 44):

The committee recommends adoption as standard of the tentative revision[3], pertaining to the inclusion of a 1 per cent chromium, 0.5 per cent molybdenum grade of material, and its incorporation into these specifications.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee A-1 recommends that the following standard be revised as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 47):

Section 8 (*b*).—Revise to read "The wall thickness at any point shall not vary more than 12½ per cent, over and under, from the average wall thickness specified."

EDITORIAL REVISIONS

Standard Specifications for Welded and Seamless Steel Pipe (A 53 – 47):

Section 3.—In the table of chemical requirements change the column heading "Lap Welded" to read "Furnace-Welded" and insert a maximum phosphorus requirement for acid-Bessemer steel of "0.11" per cent in that column.

Standard Specifications for Lap-Welded and Seamless Steel and Lap-Welded Iron Boiler Tubes (A 83 – 46):

Table I.—In the column for chemical requirements for alternate type B open-hearth iron delete the present requirements for molybdenum and copper contents and add a Footnote *c* to the table, referenced in these places, to read "Alternate type B may be specified as shown, or with a minimum copper content of 0.20 per cent, or with a molybdenum content of 0.05 to 0.15 per cent and a minimum copper content of 0.40 per cent."

Standard Specifications for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 46), and

Standard Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 46):

In the tables covering permissible variations in dimensions and weight of still tubes add a Footnote *b*, referenced after the column headings "Outside Diameter, in." and "Wall Thickness, per cent," to read "These permissible variations in outside diameter and wall thickness apply only to tubes, except internal upset tubes, as rolled or drawn, and before swaging, expanding, bending, polishing, or other fabricating operations."

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[3] 1949 Book of A.S.T.M. Standards, Part 1, p. 222.

Standard Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 47),

Standard Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 47),

Standard Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 47),

Standard Specifications for Welded Alloy-Steel Boiler and Superheater Tubes (A 249 – 47), and

Standard Specifications for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 47):

In the section covering the reverse flattening test change the requirement that the specimen be split 180 deg. from each side of the weld to read "90 deg."

Standard Specifications for Seamless Cold-Drawn Low-Carbon Steel Heat Exchanger and Condenser Tubes (A 179 – 46), and

Standard Specifications for Seamless Cold-Drawn Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 46):

In the tables covering permissible variations in dimensions revise Notes 1 and 2 as follows:

Note 1.—Revise the last sentence to read "The wall thickness of these tubes may vary plus 28 per cent, minus 0 per cent, from that specified.

Note 2.—After the word "mandrel" revise to read "the wall thickness may vary plus 35 per cent, minus 0 per cent, from that specified; and the. . . ."

REPORT OF COMMITTEE A-3
ON
CAST IRON[1]

At the 1949 Annual Meeting of the Society it was the sad duty of the Vice-Chairman to report the sudden death of E. R. Young, who had served Committee A-3 so faithfully and ably, first as secretary from 1940 to 1944 and then as chairman since 1948. The vacancy created by the demise of Mr. Young necessitated an interim election, and the officers elected to fill the unexpired terms were as follows:

Chairman, J. S. Vanick.
Vice-Chairman, C. O. Burgess.
Secretary, H. W. Stuart.

The 1950 election of officers for the ensuing term of two years resulted in the unanimous reelection of the officers mentioned above.

In addition to the meeting of Committee A-3 and several subcommittees at the 1949 Annual Meeting of the Society, the Advisory Committee and three subcommittees held meetings in Pittsburgh on March 2, 1950. The action taken at these two meetings, the Symposium on Testing Cast Iron with SR-4 Type of Gage held at the 1949 Annual Meeting, and the Symposium on the Properties, Testing and Use of Gray Iron presented at the A.S.T.M. Pacific Area National Meeting in San Francisco, constitute the major accomplishments of Committee A-3 during the past year.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee A-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Foundry Pig Iron (A 43 – 45 T), and
Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F. (A 278 – 44 T).

These recommendations were accepted by the Standards Committee on December 22, 1949, and the revised tentative specifications appear in the 1949 Book of A.S.T.M. Standards, Part 1.

REAFFIRMATION OF STANDARDS

The committee recommends that the following standards, which have stood for more than six years without revision, be reaffirmed and continued as such:

Standard Specifications for:

Cast Iron Culvert Pipe (A 142 – 38), and
Gray Iron Castings for Valves, Flanges and Pipe Fittings (A 126 – 42).

The following two standards have stood for more than six years and will be reviewed at the Annual Meeting:

Standard Specifications for:

Cast Iron Pit-Cast Pipe for Water or Other Liquids (A 44 – 41), and
Cast Iron Soil Pipe and Fittings (A 74 – 42).

NEW TENTATIVE

The committee recommends for publication as tentative the Proposed Methods of Impact Testing of Cast Iron as appended to the 1949 Report of Committee A-3.[2]

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The new tentative was accepted by the Society and appears in the 1950 Supplement to Book of Standards, Part 1.

These actions have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Pig Iron (H. W. Stuart, chairman) completed in 1949 a recommended revision of Specifications A 43 for Foundry Pig Iron, including the expansion of the silvery pig iron grades. Subsequent to letter ballot approval of Committee A-3, this revision was accepted by the Administrative Committee on Standards during 1949, as explained previously in this report.

Subcommittee XV on Impact Testing (J. T. MacKenzie, chairman) has been responsible for developing the proposed Tentative Methods of Impact Testing of Cast Iron.

Subcommittee XXII on Elevated Temperature Properties of Cast Iron (J. S. Vanick, chairman) was responsible for the 1949 revision of Specifications A 278 for Gray Iron Castings for Pressure-Containing Parts for Temperatures Up to 650 F. This action was approved by Committee A-3 and by the Administrative Committee on Standards. It was recommended in order to bring the specifications into accord with proposed action of the A.S.M.E. Boiler Code Committee.

Subcommittee XXIV on Nodular Cast Iron.—The chairman of Committee A-3 at the March 2 meeting appointed T. E. Eagan as chairman of a new Subcommittee XXIV on Nodular Cast Iron. The subcommittee membership is being organized for an initial meeting during the Annual Meeting.

Special Subcommittees.—A special subcommittee appointed at the 1949 Annual Meeting, with the aid of the A.S.T.M. staff, developed revised By-Laws for Committee A-3, which were approved by letter ballot of the committee.

Another special subcommittee appointed at the 1949 Annual Meeting studied the entire membership of Committee A-3 and re-classified the members in accordance with the policy of the Society and the conditions now existing in the cast iron industry. This re-classification has been submitted to the entire membership for their information and approval. At present the committee is composed of 36 producers, 53 consumers, 36 general interest members, and 4 consultants.

Cooperation continues between the A.F.S. and the A.S.T.M. Test Bar and Chill Test Committees and reports which may lead to tentative specifications in the future have been exchanged.

SYMPOSIUMS

The Symposium on Testing Cast Iron with SR-4 Type of Gage, held at the 1949 Annual Meeting, has been published by the Society, and it promises to become an important source of information on this method of testing throughout the cast iron industry and the engineering profession in general.[4]

The Symposium on the Properties, Testing and Use of Gray Iron at the A.S.T.M. Pacific Area National Meeting in San Francisco in October, 1949, included the following papers, which were very well received:

"Mechanical Testing and Properties of Gray Cast Iron" by C. K. Donoho, American Cast Iron Pipe Co.

"Development, Significance and Uses of Specifications for Cast Iron" by H. Bornstein, Deere and Co.

"Automotive Cast Irons, Including Brake Drums" by R. G. McElwee, Vanadium Corporation of America.

"Applications and Uses of Gray Iron Castings, Including Significance of Various Properties of Gray Cast Iron" by C. O. Burgess, Gray Iron Founders' Society.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] Issued as a separate publication, *STP No. 97*, 1949.

This symposium developed considerable discussion, and may lead to a greater interest on the West Coast in the properties of cast iron as an engineering material.[5]

This report has been submitted to letter ballot of the committee, which consists of 129 members; 93 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. S. VANICK,
Chairman.
C. O. BURGESS,
Vice-Chairman.

H. W. STUART,
Secretary.

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[5] These papers are appearing in some early 1950 issues of the magazine, *The Foundry.*

REPORT OF COMMITTEE A-5
ON
CORROSION OF IRON AND STEEL[1]

Committee A-5 on Corrosion of Iron and Steel held a meeting on June 29, 1949, during the Annual Meeting of the Society. This was the only meeting of the main committee during the year, although some of the subcommittees met on February 28, 1950, during the Spring Meeting of the Society.

Since the last report two new members have been elected and sixteen members were lost through resignation, death or other causes. The total membership of the committee is now 116, of whom 52 are classified as producers, 36 as consumers, and 28 as general interest members.

An election of officers was held with the result that T. R. Galloway was elected as chairman, and A. P. Jahn and E. F. Lundeen were elected first and second vice chairman, respectively, and H. F. Hormann as secretary.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Zinc-Coated (Galvanized) High Tensile Steel Telephone and Telegraph Line Wire (A 326 - 49 T).

Revision and Reversion to Tentative of Standard Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Telephone and Telegraph Line Wire (A 111 - 43).

Specifications A 326-49 T were prepared because of the advent of relatively high strength, low resistivity wire. Specifications A 111-43 were revised to conform to commercial developments.

These recommendations were accepted by the Standards Committee on January 12, 1950, and the new tentatives appear in the 1949 Book of A.S.T.M. Standards, Part 1.

REVISIONS OF TENTATIVES

The committee recommends that the following two tentatives, appended hereto,[2] be revised to include material coated in coils and be continued as tentative:

Tentative Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Sheets Coils and Cut Lengths (A 93 - 48 T), and

Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 - 47 T).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation of the following tentatives without change:

Tentative Specifications for:

Lead-Alloy Coating (Hot-Dip) on Iron or Steel Hardware (A 267 - 46 T).[3]—These specifications were first issued in 1942 as emergency specifications and were issued as tentative in 1944. Very little use is made of lead-alloy coated hardware at the present time and the need for these specifications in the future is a moot question. It is, therefore, desirable to retain them as tentative and to await future developments.

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.**

[2] The revised specifications were accepted by the Society and appear in the 1950 Supplement to Book of Standards, Part 1.

[3] **1949 Book of A.S.T.M. Standards, Part 1.**

Tentative Methods of:

Test for Local Thickness of Electrodeposited Coatings (A 219 - 45 T).[3]—These methods are under the joint jurisdiction of Committees A-5 and B-8. Both committees recommend that these methods remain tentative. Current research and development work by the American Electroplaters Society and A.S.T.M. on measuring thickness of coatings has not been concluded and it is desired to keep these methods tentative until this work has been completed and the results can be included.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee V on Total Immersion Tests ceased to exist actively several years ago, but circumstances prevented the preparation of a final report at that time. This report is appended hereto under the title "Final Report of Subcommittee V on Total Immersion Tests."

Subcommittee VII on Methods of Test (W. E. Buck, chairman) is studying revisions of Standard Methods of Test for Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles (A 90 - 39) to include *(1)* the 1:1 Hydrochloric Acid Stripping Method for determination of the weight of coating, *(2)* to allow the use of circular coupons as well as square coupons to agree with Specifications A 93 - 48 T and *(3)* to allow the use of rectangular test coupons (with rounded corners having a maximum radius of $\frac{1}{4}$ in.) if desired, provided they contain an area of 5.06 sq. in. (the minimum dimension of which must be 1.250 in.).

The subcommittee is studying the reactions of various zinc coatings (hot dipped, electrodeposited, heat treated, etc.) in copper sulfate solutions.

The investigation of the Hull-Strausser type dropping test is continuing.

The subcommittee is also studying various types of gages for measuring thickness of the coatings of non-magnetic materials applied to a ferous base.

Subcommittee XI on Sheet Specifications (E. F. Lundeen, chairman) prepared the revisions to Specifications A 93 and A 308, mentioned earlier in this report, to include material coated in coils. This subcommittee is studying the need for revising the coating bend test in Specifications A 93 to include heavier gages in all weights of coatings. This subcommittee is looking into the need for separate specifications for roofing sheets. As a first step, a draft of Specifications for Iron or Steel Roofing Sheets has been prepared which covers 1.25 -oz. coatings. These specifications were discussed at the Spring Meeting and it was decided to expand the draft to include other recognized weights of coating for roofing sheets.

Subcommittee XII on Wire Specifications (P. E. Chisler, chairman) prepared the revisions to Specifications A 111, and new Specifications A 326, mentioned earlier in this report. Work is presently in progress on the preparation of Specifications for Chain-Link Fence Fabric Galvanized Before Weaving. The weights of coating for fabric galvanized before weaving will go beyond that now covered in Standard Specifications for Zinc-Coated Iron or Steel Chain-Link Fence Fabric Galvanized After Weaving (A 117 - 33). After work on the specifications for fabric galvanized before weaving is completed, Specifications A 117 - 33 will be reviewed to determine the need for increasing the weight of coating requirement.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) reports that some provision for weight of coating for pieces lighter than "Structural" steel shapes should be made in Specifications A 123 - 47 which now contain only one class of coating, namely, 2.0 oz. minimum. Angles and channels of 1-in. or 1½-in. size, it is claimed, cannot take this weight of coating and Specifications A 153 for hardware are not exactly suitable for use on steel windows, for example. A study of this problem is currently being made.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Specifications A 153 - 49 are being studied as to the need for including low-alloy high-strength steel, galvanized after fabrication. The necessity of use of low-alloy steel for pole-line hardware is being recognized by many utilities in view of high labor costs of replacement. The subcommittee is continuing to gather data on weights of coating on various classes of hardware.

Subcommittee XIV on Sheet Tests (E. S. Taylerson, chairman).—The report of this subcommittee is appended hereto under the title "Report of Subcommittee XIV on Inspection of Black and Galvanized Sheets."

Subcommittee XV on Wire Tests (A. P. Jahn, chairman).—The report of this subcommittee is appended hereto under the title "Report of Subcommittee XV Covering 1949 Inspections on Field Tests of Wire and Wire Products."

Subcommittee XVI on Hardware Tests (A. Mendizza, chairman.)—The report of this subcommittee is appended hereto under the title "Report of Subcommittee XVI on Field Tests and Inspections of Bare and Metallic Coated Hardware, Structural Shapes, Plates, Bars and Their Products."

This report has been submitted to letter ballot of the committee, which consists of 116 members; 73 members returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. R. GALLOWAY,
Chairman.

H. F. HORMANN,
Secretary.

FINAL REPORT OF SUBCOMMITTEE V ON TOTAL IMMERSION TESTS

A brief summary of the activities of Subcommittee V since its organization might be of interest.

Subcommittee V on Total Immersion Tests, of A.S.T.M. Committee A-5 on Corrosion of Iron and Steel, was organized in 1919, making its first report in 1920. It was organized primarily for the purpose of conducting immersion tests on the series of copper and non-copper steels which had been exposed to the atmospheres of Ft. Sheridan, Pittsburgh and Annapolis in 1916 and 1917. The test in Calumet Mine water was installed in 1920 and those in Washington City water and Severn River brackish water in 1923. Earlier reports on these three immersion tests were published in the Committee A-5 reports which appeared in the Society's *Proceedings* for 1920, 1921, 1924, 1926, 1928 and 1930.

A paper entitled "A Critical Study of the A.S.T.M. Corrosion Data on Uncoated Commercial Iron and Steel Sheets" was presented before the Society by V. V. Kendall and E. S.Taylerson in 1929.[1]

In 1927, sea water immersion tests were installed at Portsmouth, N. H., and Key West, Fla. Several attempts by various methods have been made to analyze the data statistically to determine if there were a significant difference between copper and non-copper steel. However, when the data are considered from the standpoint of types of steel, both for No. 22 and 16 gage, the effect of other elements, and the varying effects of location, the results indicate that any beneficial effect of copper under totally immersed sea water conditions is so small as not to be of commercial importance.

In 1926, a 12-yr. exposure of some low- and high-copper seamless tubing at Portsmouth and at Key West showed no consistent effect of copper under sea water conditions.

In 1931, a comprehensive investigation of the acid corrosion test was carried out. Specimens from the steels used in the atmospheric test, on which definite life figures had been obtained, were sent to five laboratories with definite directions for acid testing. The results showed no correlation between actual atmospheric life and acid-test results. Furthermore, the results from the five laboratories were inconsistent. The conclusion from this test was as follows:

> As a result of these experiments, it is concluded that an acid test conducted by the procedure followed by the committee is not capable of consistent repetition. Further, the data from such an acid test should not be used to forecast the relative life of ordinary ferrous materials in the atmosphere.

Report prepared by V. V. Kendall and E. S. Taylerson.

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[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 29, pp. 204-219 (1929).

REPORT OF SUBCOMMITTEE XIV ON INSPECTION OF BLACK AND GALVANIZED SHEETS

The atmospheric tests of corrugated sheets at Annapolis, Md., Altoona, Pa., Brunot Island, Pittsburgh, Pa., Sandy Hook, N. J., and State College, Pa., were inspected in both April and October of 1948 and April of 1949 and 1950. Spring inspections only will be made in the future.

Table I shows failures of No. 22 gage black sheets at Annapolis (33.5 yr. exposure).

Tables II, III, IV, and V bring the data given in the 1948 report up to date (24 yr.).

The rack at Key West was inspected in 1949 and showed further development of the conditions described in former reports.

In 1948, the committee decided to check the identity of the steel base of all the Y series of galvanized corrugated sheets. The results indicate that this group of sheets is from several heats of enough difference in composition to account for the observed inconsistencies. Consequently the data obtained from this mixed series may be misleading and should be ignored.

This report has been submitted to letter ballot of Subcommittee XIV and unanimously approved.

Respectfully submitted on behalf of the subcommittee,

E. S. Taylerson,
Chairman.

TABLE I.—RECORD OF FAILURES AT ANNAPOLIS OF 22 GAGE COPPER-BEARING AND NON-COPPER-BEARING CORRUGATED BLACK SHEETS.
Sheets Exposed October 17, 1916. Inspections Made Semi-Annually From 1917 to 1950.

Group	Designation	Series	Average Analysis, per cent						Number of Sheets in Test[a]	Life of Each Failed Sheet, yr.																Number of Sheets in Test	Number of Sheets Failed	Average Life of Sheets, yr.	Group
			Carbon	Manganese	Phosphorus	Sulfur	Silicon	Copper																					
Z.....	Open-hearth Steel	B	0.032	0.32	0.063	0.042	.....	0.019	3	13.5	17.0	19.0	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	16.5	Z
O.....	Open-hearth Steel	A	0.121	0.536	0.008	0.030	0.249	0.020	12	7.0	9.0	9.0	9.0	9.0	9.5	10.0	10.0	11.0	11.0	13.0	17.0	...	...	...	...	12	12	10.4	O
B.....	Open-hearth Steel	A	0.09	0.38	0.017	0.062	0.036	0.084	3	20.0	20.5	24.0	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	21.5	B
M.....	Open-hearth Steel	B	0.093	0.419	0.013	0.041	0.011	0.185	13	20.0	20.5	23.5	23.5	23.5	24.0	24.0	24.0	24.0	24.0	24.0	24.0	25.0	...	...	...	13	13	23.4	M
B.....	Open-hearth Steel	A	0.06	0.27	0.014	0.053	0.006	0.187	9	19.5	20.0	21.5	22.0	22.5	23.0	26.0	28.0	28.5	...	...	...	...	...	...	...	9	9	23.4	B
Z.....	Open-hearth Steel	B	0.067	0.351	0.045	0.052	0.003	0.187	3	27.0	27.5	28.0	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	27.5	Z
T.....	Open-hearth Steel	B	0.083	0.313	0.009	0.020	0.005	0.227	14	19.5	19.5	19.5	21.5	21.5	21.5	23.0	23.5	23.5	23.5	24.0	24.0	24.0	24.0	...	...	14	14	22.3	T
K.....	Open-hearth Steel (Acid)	A	0.107	0.447	0.091	0.046	0.004	0.237	14	24.5	28.0	28.5	28.5	28.5	29.0	29.5	30.0	31.0	31.5	32.0	32.0	32.0	33.5	...	...	14	14	29.9	K
H.....	Open-hearth Steel	A	0.069	0.387	0.016	0.027	0.004	0.244	10	19.5	21.0	21.5	23.0	23.5	23.5	23.5	24.0	24.0	25.0	...	...	...	...	...	...	10	10	22.9	H
Z.....	Open-hearth Steel	B	0.065	0.35	0.050	0.059	0.003	0.264	2	24.5	28.0	...	...	...	...	...	...	...	...	...	...	...	...	...	...	2	2	26.3	Z
Z.....	Open-hearth Steel	B	0.073	0.42	0.052	0.054	Trace	0.641	3	28.0	29.0	33.5	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	30.2	Z
S.....	Open-hearth Iron	B	0.02	0.023	0.006	0.022	0.004	0.022	14	7.0	8.0	8.5	9.0	9.5	10.0	11.0	11.5	11.5	12.5	12.5	12.5	12.5	12.5	...	...	14	14	10.6	S
D.....	Open-hearth Iron	A	0.02	0.026	0.004	0.022	0.002	0.024	6	16.0	16.0	16.0	19.5	19.5	23.5	...	...	...	...	...	...	...	...	...	...	6	6	18.4	D
X.....	Open-hearth Iron	B	0.021	0.055	0.007	0.031	0.004	0.027	14	10.5	13.0	13.5	14.5	14.5	16.0	16.0	16.5	16.5	17.0	17.5	17.5	18.0	19.0	...	...	14	14	15.7	X
C.....	Open-hearth Iron	A	0.01	0.026	0.002	0.027	0.002	0.141	3	17.5	20.5	23.5	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	20.5	C
C.....	Open-hearth Iron	A	0.015	0.028	0.006	0.036	0.003	0.194	9	16.5	16.5	18.5	19.0	19.0	20.0	20.5	21.5	21.5	...	...	...	...	...	...	...	9	9	19.2	C
U.....	Open-hearth Iron	B	0.017	0.074	0.008	0.037	0.005	0.260	13	15.5	16.5	19.0	19.0	19.0	19.0	19.0	19.5	20.0	20.0	21.0	21.0	22.5	...	...	...	13	13	19.3	U
V.....	Open-hearth Iron	B	0.018	0.051	0.007	0.021	0.005	0.304	14	17.0	17.0	17.5	18.5	18.5	19.0	20.0	20.0	20.5	21.0	22.0	22.0	22.5	24.0	...	...	14	14	20.0	V
A.....	Bessemer Steel	A	0.038	0.386	0.089	0.040	0.007	0.014	15	15.5	16.5	16.5	17.0	17.0	17.0	18.0	19.0	19.0	19.5	19.5	19.5	19.5	19.5	20.0	...	15	15	18.2	A
Z.....	Bessemer Steel	B	0.063	0.41	0.102	0.044	0.009	0.013	3	11.5	11.5	11.5	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	11.5	Z
Z.....	Bessemer Steel	B	0.077	0.44	0.095	0.036	0.003	0.140	3	27.5	27.5	27.5	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	27.5	Z
I.....	Bessemer Steel	A	0.041	0.365	0.097	0.068	0.008	0.252	15	24.0	26.0	26.5	26.5	26.5	26.5	27.0	28.0	28.0	29.0	29.5	29.5	31.0	31.5	33.5	...	15	15	28.2	I
Z.....	Bessemer Steel	B	0.058	0.37	0.093	0.043	0.009	0.269	3	28.0	29.5	31.0	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	29.5	Z
Z.....	Bessemer Steel	B	0.055	0.38	0.110	0.046	.....	0.527	3	32.0	32.5	*	...	...	...	...	...	...	...	...	...	...	...	...	...	3	2	....	Z
Y.....	Wrought Iron	B	0.03	0.055	0.139	0.021	0.218	0.020	3	18.0	19.5	23.5	...	...	...	...	...	...	...	...	...	...	...	...	...	3	3	20.3	Y
E.....	Wrought Iron	A	0.033	0.034	0.114	0.021	0.134	0.283	10	19.5	20.5	21.0	21.0	23.0	23.0	23.0	23.0	24.0	25.0	...	...	...	...	...	...	10	10	22.3	E
							Total......		214																Total...	214	213		

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* Indicates unfailed sheet.

[a] The number of sheets in the test indicated in this table is less than the number shown in earlier reports because certain sheets were discarded due to mechanical damage.

TABLE II.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.

Exposed at Altoona, Pa., on April 22, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Uncoated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			Years To Perforation
	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	
R (T)	....	....	....	4.3	12.0	21.0	3.3	9.5	18.5	3.3	5.0	14.5	2.3	4.3	14.5	....	....	....	....	....	....	4.3
(B)				4.5	11.5	21.5	3.3	9.0	16.5	3.3	7.0	17.0	2.3	4.5	16.0							
V (T)	4.3	11.0	*	4.3	9.0	[b]	3.3	7.0	17.5	2.3	7.0	14.5	2.3	5.2	13.5	1.4	3.3	9.0	....	....	....	4.3(V)
(B)	4.3	7.0	*(U)	4.8	9.0	17.5	4.3	6.8	19.0	2.5	7.0	15.0	2.3	4.5	15.0	2.3	3.3	13.0				4.3(U)
N (T)	....	....	....	4.3	8.5	22.5	3.3	8.5	20.0	3.3	5.5	17.0	2.3	4.5	15.0	1.4	2.8	13.0	2.0	3.3	7.0	7.5
(B)				3.5	12.0	24.0	3.5	8.0	22.0	3.3	5.9	19.0	2.3	4.5	[b]	1.4	3.3	15.0	2.3	3.3	11.0	
T (T)	4.5	10.5	*	5.5	11.5	22.5	3.5	7.5	20.0	2.3	5.0	18.5	2.3	5.0	15.0	1.4	2.8	13.5	2.0	2.8	9.0	8.5
(B)	4.8	9.0	*	6.3	11.5	*	3.5	7.0	22.0	2.5	5.2	17.0	2.3	4.3	17.0	2.3	3.0	15.0	2.3	3.3	11.0	
Y (T)	4.3	11.5	17.5	3.5	11.5	15.5	3.3	8.5	17.5	2.5	5.0	14.0	2.3	3.3	14.0	2.3	3.5	4.3	1.4	3.5	10.0	2.0
(B)	4.5	13.7	16.5	3.3	9.5	17.5	4.3	9.0	19.0	3.3	5.2	17.0	2.5	4.3	15.0	2.3	3.3	8.5	2.3	3.3	[b]	
S (T)	4.3	12.0	*	4.3	12.0	22.0	4.3	8.5	20.5	3.0	4.5	19.0	2.3	4.0	17.0	2.0	2.8	13.5	2.0	3.0	7.0	9.0
(B)	4.3	12.5	*	4.8	14.5	24.0	4.3	8.5	23.0	3.3	6.3	20.5	3.0	4.3	19.0	2.3	2.8	15.0	2.3	3.0	10.5	
C (T)	....	....	....	4.0	10.5	*	3.3	8.5	22.5	2.5	6.5	21.5	2.3	4.5	19.0	1.4	3.5	[b]	....	....	....	[b]
(B)				5.8	13.5	*	4.3	10.0	24.0	3.3	6.5	19.0	2.5	4.3	19.5	1.4	3.0	[b]				
K (T)	4.3	12.5	*	4.3	11.5	*	4.3	7.5	24.0	3.3	5.2	21.5	2.5	4.3	[b]	2.3	3.0	[b]	2.3	2.8	10.5	13.0
(B)	4.3	11.5	*	4.5	11.5	*	4.3	8.5	*	3.3	5.0	*	3.3	4.3	21.0	2.3	3.0	21.0	2.3	3.0	10.5	
W (T)	....	....	....	5.2	10.5	21.5	3.5	6.5	19.0	3.3	5.0	15.0	2.3	4.0	14.5	....	....	....	....	....	....	5.0
(B)				5.2	12.0	23.0	4.3	7.5	19.5	3.3	5.5	17.0	2.3	4.0	15.0							
Average	4.4	11.1		4.6	11.2		3.8	8.1		3.0	5.7		2.4	4.3		1.9	3.1		2.1	3.1		

* Data not yet available.

[a] Coating weight is "oz. per sq. ft." of sheet (both sides).

[b] Blown away or discarded due to mechanical damage.

TABLE III.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at Brunot Island, Pittsburgh, Pa., on April 21, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Uncoated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			Years To Perforation
	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	
R (T)	5.3	13.5	*	5.3	13.0	19.5	4.3	14.4	20.0	3.5	8.5	17.5	3.3	6.7	15.5	....	....	....	....	....	....	2.0
(B)	5.5	14.4	*	5.8	13.5	24.0	4.5	13.0	18.5	3.8	9.5	17.5	3.3	6.7	17.0							
V (T)	5.5	13.5	*	4.0	12.5	19.5	4.3	9.5	22.0	3.3	11.3	18.5	3.3	7.5	17.5	2.0	4.5	15.5	....	....	....	2.3(V)
(B)	5.0	12.0	*(U)	5.5	13.0	23.0	4.8	10.5	20.6	3.3	10.5	18.5	3.3	6.7	18.5	2.3	4.5	13.5				2.5(U)
N (T)	....	....	....	5.3	12.5	*	4.5	11.5	*	3.3	9.5	19.5	2.5	5.5	19.0	2.0	3.5	16.5	2.3	3.5	10.5	3.0
(B)				5.3	15.5	*	4.8	11.5	*	3.8	8.5	22.5	3.3	6.0	19.5	2.0	3.5	18.0	2.3	4.5	10.5	
T (T)	5.3	12.5	*	6.3	13.5	*	4.0	11.0	*	3.3	6.7	*	2.5	6.5	23.0	2.3	4.3	19.0	2.0	3.0	10.5	9.0
(B)	5.8	13.5	*	8.5	15.5	*	4.5	11.0	*	3.3	8.5	22.0	3.3	6.0	24.0	2.3	4.5	19.5	2.3	4.3	12.5	
Y (T)	4.8	14.4	11.5	4.5	11.5	19.0	3.5	11.0	17.0	3.5	6.7	15.5	3.3	4.8	15.5	2.0	4.3	20.0	2.0	3.8	19.0	2.0
(B)	5.5	16.5	11.5	5.5	14.0	20.6	4.5	16.5	14.0	3.3	8.3	17.5	3.3	6.0	13.0	2.3	5.0	20.0	2.3	4.5	[b]	
S (T)	4.3	15.5	*	5.3	15.0	*	4.5	10.5	*	3.5	7.4	19.0	3.3	5.5	21.5	2.3	4.8	14.5	2.3	3.3	4.0	3.3
(B)	5.3	15.5	*	7.0	18.5	24.0	5.3	12.5	*	3.8	7.4	23.0	3.3	6.0	22.5	2.3	3.5	18.0	2.3	4.0	4.3	
C (T)	....	....	....	4.3	12.0	*	4.0	11.5	*	3.3	10.5	*	3.3	6.3	*	2.0	4.3	*	....	....	....	12.5
(B)				8.5	18.5	*	4.8	11.0	*	3.3	10.5	*	3.3	6.8	*	2.3	4.5	22.5				
K (T)	4.8	14.0	*	5.5	15.0	*	4.3	10.5	*	4.0	8.3	*	3.3	5.0	*	2.3	3.5	*	2.3	3.0	14.5	16.5
(B)	5.3	15.5	*	7.3	16.5	*	5.8	12.5	*	4.3	8.5	*	3.3	6.5	*	2.3	4.0	*	2.3	3.5	14.5	
W (T)	....	....	....	5.5	12.5	*	4.5	9.5	21.5	3.3	7.3	18.0	3.3	5.5	17.5	....	....	....	....	....	....	3.0
(B)				6.3	15.5	*	5.8	12.5	22.0	3.3	7.3	13.0	3.3	6.0	19.5							
Average	5.2	14.3		5.9	14.4		4.6	11.7		3.5	8.6		3.2	6.1		2.2	4.2		2.2	3.7		

* Data not yet available.

[a] Coating weight is "oz. per sq. ft." of sheet (both sides).

[b] Blown away or discarded due to mechanical damage.

TABLE IV.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at Sandy Hook, N. J., on May 20, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Un-coated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			Years To Perforation
	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	
R (T)	13.4	*	*	12.4	*	*	8.4	*	*	6.4	17.9	*	7.4	14.4	*	....	....	....	....	....	....	5.9
(B)	13.4	*	*	12.9	*	*	9.4	21.9	*	7.4	19.4	*	6.9	15.9	*							
V (T)	12.4	24.0	*	10.9	24.0	*	9.9	19.4	*	7.4	19.9	*	6.4	19.4	*	5.4	13.4	*	....	....	....	4.9(V)
(B)	10.9	*	*(U)	12.4	23.0	*	10.4	19.4	*	6.9	20.4	*	7.4	15.4	*	4.5	11.0	*				5.4(U)
N (T)	....	....	....	11.4	*	*	9.4	*	*	7.4	18.4	*	6.9	15.4	*	4.5	11.4	*	4.5	11.4	24.0	5.9
(B)				10.4	*	*	9.9	20.9	*	7.4	17.4	*	5.9	15.4	*	4.5	11.0	*	6.4	12.0	*	
T (T)	12.4	*	*	15.9	*	*	9.9	23.0	*	6.9	17.9	*	5.9	15.9	*	4.5	13.4	*	4.5	13.4	20.9	6.4
(B)	12.4	24.0	*	16.4	*	*	9.9	19.4	*	6.9	14.4	*	5.4	15.4	*	4.5	11.0	*	5.4	12.0	*	
Y (T)	10.9	*	*	11.4	23.0	*	8.4	*	*	7.4	17.4	*	6.4	16.4	*	4.5	11.0	*	4.5	12.0	24.0	3.4
(B)	12.4	*	*	13.4	*	*	8.4	24.0	*	8.4	17.9	*	6.9	13.4	*	5.4	13.4	*	4.5	11.4	*	
S (T)	10.4	*	*	11.9	*	*	9.9	21.4	*	7.4	15.9	*	5.9	12.9	*	4.5	9.9	*	4.5	9.4	20.9	9.9
(B)	11.4	*	*	13.4	*	*	12.4	23.0	*	9.4	16.9	*	7.4	15.4	*	4.9	9.4	*	5.4	9.4	*	
C (T)	....	....	....	11.4	*	*	8.4	*	*	7.4	19.4	*	6.9	15.4	*	4.5	12.4	*	....	....	....	10.4
(B)				18.4	*	*	9.4	24.0	*	7.4	19.4	*	7.4	14.4	*	4.5	11.4	*				
K (T)	10.9	*	*	11.4	*	*	10.4	20.9	*	8.4	15.9	*	7.4	13.9	*	4.5	9.4	*	4.5	10.0	24.0	12.4
(B)	10.4	*	*	12.9	*	*	11.4	23.0	*	8.4	17.4	*	7.4	13.9	*	6.4	10.4	*	4.9	10.0	*	
W (T)	....	....	....	13.4	*	*	11.4	23.0	*	8.4	17.4	*	7.4	14.4	*	....	....	....	....	....	....	4.9
(B)				14.4	*	*	11.4	21.9	*	7.4	19.4	*	6.4	16.4	*							
Average	11.8			13.1			9.9			7.6	17.9		6.8	15.2		4.8	11.3		4.9	11.1		

* Data not yet available.
[a] Coating weight is "oz. per sq. ft." of sheet (both sides).

TABLE V.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at State College, Pa., on April 23, 1926.

Sheet	No. 16 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.5 oz. per sq. ft.[a]			No. 22 Gage, 2.0 oz. per sq. ft.[a]			No. 22 Gage, 1.5 oz. per sq. ft.[a]			No. 22 Gage, 1.25 oz. per sq. ft.[a]			No. 22 Gage, 0.75 oz. per sq. ft.[a]			No. 28 Gage, 0.75 oz. per sq. ft.[a]			No. 22 Gage, Uncoated
	Years To			Years To			Years To			Years To			Years To			Years To			Years To			Years To Perforation
	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	
R (T)	24.0	*	*	*	*	*	19.0	*	*	17.5	*	*	14.5	*	*	....	....	....	....	....	....	*
(B)	*	*	*	*	*	*	20.0	*	*	16.5	*	*	15.0	*	*							
V (T)	*	*	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	23.0	*				24.0(V)
(B)	24.0	*	*(U)	*	*	*	*	*	*	15.5	*	*	16.5	*	*	10.5	24.0	*	....	....	....	*(U)
N (T)	....	....	....	20.5	*	*	22.5	*	*	17.5	*	*	12.0	*	*	6.5	20.5	*	11.0	23.0	*	*
(B)				*	*	*	22.5	*	*	15.5	*	*	13.5	*	*	9.0	23.0	*	11.5	*	*	
T (T)	*	*	*	*	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	*	*	9.0	24.0	*	*
(B)	*	*	*	*	*	*	23.0	*	*	14.5	*	*	13.5	*	*	10.5	22.5	*	12.0	*	*	
Y (T)	21.5	*	*	22.5	*	*	19.5	*	*	17.5	*	*	14.5	*	*	10.0	22.5	*	9.5	24.0	*	*
(B)	22.5	*	*	*	*	*	19.0	*	*	18.5	*	*	14.5	*	*	10.5	*	*	13.0	*	*	
S (T)	22.0	*	*	*	*	*	*	*	*	19.0	*	*	14.5	*	*	10.5	21.5	*	11.5	22.0	*	*
(B)	22.5	*	*	*	*	*	*	*	*	19.5	*	*	15.5	*	*	11.0	21.5	*	11.5	21.5	*	
C (T)	....	....	....	22.5	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	24.0	*	....	....	....	*
(B)				*	*	*	21.5	*	*	18.5	*	*	14.5	*	*	10.0	24.0	*				
K (T)	23.0	*	*	*	*	*	22.5	*	*	17.5	*	*	14.5	*	*	11.5	24.0	*	10.5	23.0	*	*
(B)	24.0	*	*	*	*	*	*	*	*	21.0	*	*	16.5	*	*	11.5	22.5	*	11.5	21.0	*	
W (T)	....	....	....	*	*	*	*	*	*	16.5	*	*	13.5	*	*	....	....	....	....	....	....	*
(B)				*	*	*	*	*	*	15.0	*	*	14.5	*	*							
Average										17.0			14.6			10.0			11.1			

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* Data not yet available.

[a] Coating weight is "oz per sq. ft." of sheet (both sides).

REPORT OF SUBCOMMITTEE XV ON FIELD TESTS OF WIRE AND WIRE PRODUCTS

(COVERS 1949 INSPECTIONS)

This report summarizes the results of the atmospheric corrosion tests on wire and wire products through 1949. Detailed results are reported in the odd-numbered years, and reference to these earlier reports of Committee A-5 on Corrosion of Iron and Steel, which appeared in the Society's *Proceedings* for 1939, 1941, 1943, 1945, 1947, and 1949, will be helpful in studies of these exposure test results.

The materials under test are mostly bare steel and zinc-coated wires and wire products, but copper-covered and lead-coated steel wires and chromium and chromium-nickel steel wires are also included. The coatings are expressed in weight terms of ounces per square foot of surface. For ready comparisons of coating thickness, 1 oz. per sq. ft. of surface may be considered as averaging 0.0017 in. thick for zinc coatings, 0.0010 in. thick for lead[1] coatings, and 0.0013 in. thick for copper coatings.

Specimens of wires and wire products have been exposed for about 13 yr. at eleven locations. The location of these sites and their general classification regarding atmospheres are as follows:

Test Location	General Type of Atmosphere	Remarks
Pittsburgh, Pa.	Severe Industrial	On Brunot Island in the Ohio River about 2 miles west of the city.
Bridgeport, Conn.	Industrial	In the city.
Sandy Hook, N. J.	Seacoast	About 300 yd. from Atlantic Ocean.
Santa Cruz, Calif.	Seacoast	About 3 miles from Pacific Ocean.
State College, Pa.	Rural	Central Pennsylvania.
Lafayette, Ind.	Rural	Wabash River Valley.
Ames, Iowa	Rural	Central Iowa.
Manhattan, Kans.	Rural	Kansas River Valley.
Ithaca, N. Y.	Rural	Central New York.
College Station, Tex.	Rural	About 120 miles inland.
Davis, Calif.	Rural	About 80 miles inland.

At each location, more than 900 specimens were exposed. These included short lengths of wire (42 in. long) and wire strand at all locations; farm field fence at nine sites; barbed wire at eight locations; and chain link fence at eight locations.

The extent of corrosion is being measured by visual examination of all specimens at the test site and by tension tests on the unfabricated wire specimens. Loss of weight tests on lengths of zinc-coated wire were completed earlier in this corrosion study and the results were reported in the above-mentioned *Proceedings*. During 1949 the Pittsburgh, Bridgeport, Sandy Hook, and State

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[1] The lead-coated wires in these tests were lead coated over a bonding coat of zinc. See *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 87 (1943) for analyses of lead coatings.

TABLE A.—CORROSION CONDITIONS OF ZINC-COATED UNFABRICATED WIRES AFTER APPROXIMATELY 13 YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R + Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pin holes.

Location	Coating Group, oz. per sq. ft. of Surface										
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 to 0.85	0.80 to 1.00	1.20 to 1.35	1.60 to 1.85	2.00 or more
Pittsburgh, Pa.	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R	100R
Sandy Hook, N. J.	100R	100R	100R	100R	100R	100R	100R	100R	95R	5R	G
Bridgeport, Conn.	100R	100R	100R	100R	100R	100R	100R	100R	99R	15R	GY
State College, Pa.	100R	100R	100R	100R	75R	60R	40R	10R	GY	G	G
Lafayette, Ind.	100R	100R	100R	100R	100R	99R	99R	60R	GY	G	G
Ames, Iowa	100R	100R	100R	30R	40R	2R	1R	G	G	G	G
Manhattan, Kans.	40R	35R	1R	1R	1R	G	G	G	G	MG	G
Ithaca, N. Y.	100R	100R	100R	100R	100R	85R	60R	20R	GY	G	MG
Santa Cruz, Calif.	20R	20R	5R	5R	1R	1R	1R	G	....	G	MG
College Sta., Tex.	90R	55R	10R	10R	1R	5R	1R	1R	G	G	G
Davis, Calif.	10R	10R	G	G	G	G	G	G	....	MG	MG

TABLE B.—CORROSION CONDITIONS OF UNFABRICATED WIRES CARRYING COATINGS OTHER THAN ZINC AFTER APPROXIMATELY 13 YEARS' EXPOSURE.

R = rust; Y = yellowed; G = gray; M = metallic; MG and GY = intermediate states; D = dark to exclusion of better observation; SR and SY = superficial R and Y; R + Y = indistinguishable proportions of R and Y; Gn = green; Br = brown; PHR and PHY = rust or yellowed in pin holes.

Location	Coating Groups and Corrosion-Resistant Groups				
	Copper-Covered Wires[a]	Lead-Coated Wires[b]	12 to 14 per cent Chromium Cold Drawn	12 to 14 per cent Chromium Air Quenched	18 per cent Chromium 8 per cent Nickel
Pittsburgh, Pa.	D	D	D	D	D
Sandy Hook, N. J.	Gn	100R+Y	Y	Y	GY
Bridgeport, Conn.	Gn	GY	GY	GY	MG
State College, Pa.	Gn	100R+Y	MG	MG	M
Lafayette, Ind.	Gn	80PHR	M	M	M
Ames, Iowa	Br	25R	SY	SY	SY
Manhattan, Kans.	Br	5R	MG	MG	M
Ithaca, N. Y.	Br	10R+Y	SY	SY	M
Santa Cruz, Calif.	Br	30SR	75SR	35SR	MG
College Sta. Tex.	Br	25R+Y	99R+Y	80R+Y	5R+Y
Davis, Calif.	Br	30SR	50SR	20SR	MG

[a] Weight of coating, 7 to 9 oz. per sq. ft. of surface.
[b] Weight of coating, 1.4 to 1.9 oz. per sq. ft. of surface.

TABLE F.—CORROSION CONDITIONS OF WIRE STRAND AFTER APPROXIMATELY 13 YEARS' EXPOSURE.

R = rust; Y = yellowed; R+Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz. Zinc per sq. ft. of Surface				Lead Coated[a]
	0.69 to 0.93	1.03 to 1.17	1.32 to 1.51	1.71 to 2.47	
Pittsburgh, Pa.	100R	100R	100R	100R	100R+Y
Sandy Hook, N. J.	35R	20R	5R	G	100R+Y
Bridgeport, Conn.	45R	5R	GY	GY	5R
State College, Pa.	G	G	G	G	100R+Y
Lafayette, Ind.	G	G	G	G	100PHR
Ames, Iowa	G	G	G	G	45R
Manhattan, Kans.	G	MG	MG	MG	10R
Ithaca, N. Y.	G	G	G	M	15R
Santa Cruz. Calif.	G	G	G	MG	60SR
College Sta., Tex	G	G	G	G	10R
Davis, Calif.	G	MG	MG	MG	10R

[a] Weight of coating, 1.96 oz. per sq. ft. of surface.

TABLE C.—CORROSION CONDITIONS OF FARM FIELD FENCE AFTER APPROXIMATELY 13 YEARS' EXPOSURE.

R = rust; Y = yellowed; R + Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz. Zinc per sq. ft. of Surface							Copper-Covered[a]	Lead-Coated[b]	Chromium-Nickel Steel
	0.20 to 0.30	0.25 to 0.35	0.35 to 0.45	0.45 to 0.55	0.50 to 0.60	0.60 to 0.70	0.70 or more			
State College, Pa	100R	100R	100R	100R	70R	60R	G	Gn	100R+Y	MG
Lafayette, Ind	100R	100R	100R	100R	95R	65R	5R	Gn	50PHR	M
Ames, Iowa	100R	100R	85R	30R	10R	G	G	Br	25R	SY
Manhattan, Kans	25R	30R	GY	GY	G	G	MG	Br	15R	MG
Ithaca, N. Y.	100R	99R	90R	75R	65R	40R	1R	Gn	5R	MG
Santa Cruz, Calif.	25R	25R	10R	10R	1R	5R	1R	Br	40SR	50R
College Sta., Tex.	60R	50R	20R	5R	5R	5R	G	Br	15R	10SR
Davis, Calif.	25R	25R	1R	G	G	G	MG	Br	40SR	20SR

[a] Weight of coating, 3.97 oz. per sq. ft. of surface.
[b] Weight of coating, 1.83 oz. per sq. ft. of surface.

TABLE D.—CORROSION CONDITIONS OF BARBED WIRE AFTER APPROXIMATELY 13 YEARS' EXPOSURE.

R = rust; Y = yellowed; R + Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown.

Location	Coating Group, oz. Zinc per sq. ft. of Surface							Copper Covered[a]	Lead Coated[b]
	0.20 to 0.30	0.35 to 0.45	0.50 to 0.60	0.70 to 0.85	0.80 to 1.00	1.60 to 1.80	2.00 or more		
State College, Pa	100R	100R	60R	G	GY	G	G	Gn	....
Lafayette, Ind.	100R	100R	99R	75R	10R	5R	5R	Gn	50PHR
Ames, Iowa	100R	85R	5R	G	G	G	G	Br	20R
Manhattan, Kans.	35R	5R	GY	MG	MG	MG	MG	Br	5PHY
Ithaca, N. Y.	100R	100R	60R	1R	G	G	M	Gn	10R
Santa Cruz, Calif.	25R	10R	5R	G	G	G	MG	Br	30SR
College Sta., Tex.	70R	15R	5R	G	G	G	G	Br	3R
Davis, Calif.	10R	G	G	MG	MG	MG	MG	Br	25SR

[a] Weight of coating, 3.97 oz. per sq. ft. of surface.
[b] Weight of coating, 1.83 oz. per sq. ft. of surface.

TABLE E.—CORROSION CONDITIONS OF CHAIN LINK FENCE AFTER APPROXIMATELY 13 YEARS' EXPOSURE.

R = rust; Y = yellowed; R + Y = indistinguishable proportions of R and Y; G = gray; M = metallic; MG and GY = intermediate state; PHR, PHY = pin hole rusting or yellowing; SR, SY = superficial rust or yellowing; Gn = green; Br = brown; D = dark (dirt or soot) to the exclusion of a better observation.

Location	Coating Group, oz. Zinc per sq. ft. of Surface					Copper Covered[c]	Lead Coated[d]	Corrosion Resistant
	0.29 to 0.52[a]	1.13 to 1.22[a]	2.09 to 2.92[a]	1.40 to 1.97[b]	4.21 or more[b]			
Pittsburgh, Pa.	100R	100R	100R	100R	100R	..	D	D
Sandy Hook, N. J.	100R	85R	G	100R	GY	Gn	50R+Y	MG
Bridgeport, Conn.	100R	100R	G	80R	GY	Gn	Y	MG
State College, Pa	90R	GY	G	GY	G	Gn	100R+Y	MG
Lafayette, Ind.	100R	5R	G	GY	G	Gn	100PHR	M
Ithaca, N. Y.	95R	GY	MG	GY	GY	Br	10R	MG
Santa Cruz, Calif.	10R	MG	MG	MG	MG	Br	35SR	MG
Davis, Calif.	5R	MG	MG	MG	MG	Br	35SR	MG

[a] Coated before weaving.
[b] Coated after weaving.
[c] Weight of coating, 8.52 oz. per sq. ft. of surface.
[d] Weight of coating, 1.75 oz. per sq. ft. of surface.

College specimens were inspected in the spring by a traveling committee. Inspections were made in the fall of 1949 at the other test sites by the university people in charge of the test plots.

The summarized inspection records of corrosion through 1949 are shown in Tables A and B for unfabricated wire specimens, Table C for farm field fence, Table D for barbed wire, Table E for chain link fence, and Table F for wire strand.

Tensile strength tests were made on 113 selected specimens during 1949. Of these specimens 18 were taken from Sandy Hook, 26 from Bridgeport, and 69 from Pittsburgh. These tensile strength data do not show any noticeable change from the rate of tensile strength loss previously reported.

Respectfully submitted on behalf of the Wire Test Inspection Committee,

A. P. Jahn,
Chairman.

REPORT OF SUBCOMMITTEE XVI ON FIELD TESTS AND INSPECTIONS OF BARE AND METALLIC-COATED HARDWARE, STRUCTURAL SHAPES, PLATES, BARS, AND THEIR PRODUCTS

The data obtained in the routine annual inspection of two of the three remaining test locations have been assembled in Table I. These cover the period from April, 1948 to April, 1950. In view of the slow progress of corrosion of the specimens still remaining on exposure, it has been decided to make only annual spring inspections. This practice began in 1948. No inspection has been made at Key West since April 1947. The last tabulated report for this location appears in the 1948 Report.[1]

Altoona and Brunot Island, Pa.—All tests at these locations have been discontinued. The last report is tabulated in the 1944 Report.[2]

State College, Pa., and Sandy Hook, N. J.—Only zinc-, aluminum-, and lead-coated specimens remain at these two locations. No appreciable changes have occurred at either site since the 1948 Report.[1] The data for the last inspection (1950) are, in general, comparable to those obtained in 1947. Minor changes, in some instances indicating improved conditions, are probably more the result of variations amongst individual inspectors than real changes in appearance.

Respectfully submitted on behalf of the subcommittee,

A. Mendizza,
Chairman.

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[1] Report of Subcommittee XVI, *Proceedings*, Am. Soc. Testing Mats., Vol., 48, p. 119 (1948).

[2] Report of Subcommittee VIII, *Proceedings*, Am. Soc. Testing Mats., Vol. 44, p. 92 (1944).

TABLE I.—REPORT OF INSPECTIONS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC.; EXPOSED TO ATMOSPHERE ON TEST RACKS AT TWO LOCATIONS.

Abbreviations and symbols used: 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; R*, rust spots widely distributed; Y, yellow or orange yellow rust; D, black or dark surface films (soot or dirt); Tr, trace; X, 100 per cent rust listed in previous reports; TH, threads; a, samples mixed; b, average of nipple and ell. Blank spaces indicate samples removed in 1942.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.			Exposure at Sandy Hook, N. J.		
		4-22-48 990 Weeks	4-27-49 1042 Weeks	4-26-50 1094 Weeks	4-21-48 999 Weeks	4-26-49 1052 Weeks	-25-50 1104 Weeks
3 BY 3 BY 5/16-IN. PLAIN STEEL ANGLES							
Hot-dip galvanized	2.52	0	0	TrY	5Y	5Y	TrR-10Y
Hot-dip aluminum	0.89	0D	0D	0D	0	TrY-D	TrY-D
Electric Sherardized	1.38	TrR-100Y	TrR-100Y	TrR-100R*	...	...	...
Lead coated	...	TrR-98R*	90R*	7R-85R*	...	...	...
3 BY 3 BY 5/16-IN. COPPER-BEARING STEEL ANGLES							
Hot-dip galvanized	2.88	6Y	3Y	5Y	5Y	5Y	TrR-10Y
Hot-dip aluminum	0.82	0D	0D	0D	0	TrY-D	TrY-D
Electric Sherardized	1.44	3R-97Y	3R-97Y	7R-90R*	...	...	...
Lead coated	...	TrR-70R*	5R-90R*	TrR-90R*	...	...	...
2 BY 1½ BY 5/16-IN. PLAIN STEEL ANGLES							
Hot-dip galvanized	3.24	10Y	5Y	5Y	TrR-5Y	3R-10Y	TrR-20Y
Hot-dip aluminum	0.75	0	0	0	0D	5Y	5Y
Lead coated	...	80R*	1R-70R*	90R*	60R*	TrR-60R*	TrR-80R*
2 BY 1½ BY 5/16-IN. COPPER-BEARING STEEL ANGLES							
Hot-dip galvanized	2.75	0	TrY	TrY	5Y	3R-10Y	TrR-20Y
Hot-dip aluminum	0.50	0	0	0	0	5Y	5Y
Lead coated	...	80R*	5R-80R*	TrR-92R*	...	...	...
2-IN. DIAMETER TUBULAR GOODS							
Electrogalvanized	1.58	0	0	TrY	...	...	...
Hot-dip galvanized	2.51	3Y	10Y	17Y	10Y	20Y	TrR-25Y
Hot-dip aluminum	4.73	0D	0	0	0D	TrY	5Y-D
Gas Sherardized	0.66	30R-70Y	30R-70Y	30R-70Y	...	...	...
Lead coated	...	80R*	2R-90R*	2R-90R*	100R*	5R-80R*	10R-90R*
1-IN. DIAMETER TUBULAR GOODS							
Electrogalvanized	1.62	0	0	0	...	...	...
Hot-dip galvanized	1.83	100Y	90Y	TrR-97Y	...	...	...
Hot-dip aluminum	1.83	0D	0D	0D	...	...	...
Electric Sherardized	0.92	20R-80Y	30R-70Y	55R-45Y	...	...	...
Gas Sherardized	0.72	40R-50Y	50R-50Y	40R-60Y	...	...	...
Calorized	...	TrY-D	10R*-D	5R*	...	...	...
Lead coated	...	60R*	1R-90R*	70R*	...	...	...
Amaloy	...	60R*	1R-90R*	90R*	...	...	...

FORGED STEEL TOWER FITTINGS							
Hot-dip galvanized	2.67	15Y	TrR-60Y	50Y	...	...	...
Hot-dip galvanized	1.53	60Y			...	...	...
Hot-dip aluminum	...	0D	0	0	...	...	...
Electric Sherardized	0.50	25R-75Y	35R-65Y	25R-75Y	...	...	...
¾-IN. NIPPLES AND ELLS							
Electrogalvanized M1	1.60	0	TrR	TrR-TrY	...	...	...
Electrogalvanized C1	2.19	10R-10Y	10R	10R-5Y	...	...	...
Electrogalvanized M1	2.65	30R	20R	R in TH-50R-25Y	...	...	...
Electrogalvanized C1	1.25	20R	25R	45R-30Y	...	...	...
Hot-dip galvanized M1	1.82	80Y	90Y	100Y	30R-70Y	55R-60Y	75R-25Y
Hot-dip galvanized C1	1.44	80Y	70Y	97Y			
Hot-dip aluminum M1	3.20	R in TH	R in TH	R in TH-5Y	R in TH	R in TH	R in TH-25Y
Hot-dip aluminum C1	2.80	R in TH	R in TH	R in TH			
Electric Sherardized M1	0.75	10R-90Y	10R-90Y	5R-95Y	100R*	TrR-90R*	10 R-9
Amaloy M1 and C1	...	R in TH-100R*	15R-85R*	R in TH-10R-90R*			
TOWER CLAMPS							
Hot-dip galvanized M1	2.00	60Y	90Y	15Y	95Y	7R-93Y	5R-90Y
Hot-dip galvanized C1	2.38	10Y	25Y		TrR-92Y		
Hot-dip aluminum M1	3.16	0	0D	0D	0D	0D	0D
Hot-dip aluminum C1	2.51						
1½ BY 1½ BY ⅛-IN. PLAIN STEEL ANGLES							
Hot-dip galvanized	3.16	0	0	0	5Y	TrR-8Y	TrR-15Y
Hot-dip aluminum	0.89	0D	0	0D	0D	0D	0D
Lead coated	...	5R-70R*	5R-95R*	5R-80R*	80R*	5R-90R*	10R-85R*
1½ BY 1½ BY ⅛-IN. COPPER-BEARING STEEL ANGLES							
Hot-dip galvanized	3.20	5Y	5Y	TrY	5Y	TrR-12Y	TrR-10Y
Hot-dip aluminum	1.11	0D	0D	0D	0	0D	0D
Lead coated	...	5R-60R*	5R-90R*	TrR-90R*	60R*	5R-55R*	5R-70R*
NO. 14 GAGE BY 2½-IN. PLAIN STEEL FLATS							
Hot-dip galvanized	3.16	...	...	...	5R-15Y	5R-15Y	10R-25Y
Hot-dip aluminum	0.89	...	...	...	0	0D	0D
Lead coated	...	...	...	...	70R*	5R-60R*	5R-80R*
NO. 14 GAGE BY 2½-IN. COPPER-BEARING STEEL FLATS							
Hot-dip galvanized	3.20	5Y	5Y	5Y	...	...	...
Hot-dip aluminum	0.47	0	0	0	...	...	...
Lead coated	...	60R*	TrR-40R*	75R*	...	...	...

TABLE I.—REPORT OF INSPECTIONS ON HARDWARE, STRUCTURAL SHAPES, TUBULAR GOODS, ETC.; EXPOSED TO ATMOSPHERE ON TEST RACKS AT TWO LOCATIONS.—*Continued.*

Abbreviations and symbols used; 0, no rust; 5, 10, 25, 100, approximate percentages; R, rusting of base metal; R*, rust spots widely distributed; Y, yellow or orange yellow rust; D, black or dark surface films (soot or dirt); Tr, trace; X, 100 per cent rust listed in previous reports; TH, threads; a, samples mixed; b, average of nipple and ell. Blank spaces indicate samples removed in 1942.

Coating	Weight, oz. per sq. ft. of surface	Exposure at State College, Pa.			Exposure at Sandy Hook, N. J.		
		4-22-48 990 Weeks	4-27-49 1042 Weeks	4-26-50 1094 Weeks	4-21-48 999 Weeks	4-26-49 1052 Weeks	4-25-50 1104 Weeks
			KNOCKOUT BOXES				
Hot-dip galvanized	1.56	20Y	25Y	25Y	5R-95Y	7R-93Y	15R-85Y
Electric Sherardized	0.28	100Y	100Y	100Y	...	...	...
Amaloy	...	50R*	5R-80R*	5R-95R*	99R*	TrR-95R*	15R-85R*
Hot-dip aluminum	1.84	0	0D	0D	0	7Y	5Y-D
			BULLDOG CLAMPS				
Hot-dip galvanized	1.88	...	...	...	20R-80Y	75R-25Y	80R-20Y
Hot-dip aluminum	1.35	0D	TrR	TrR-TrY	0	TrY	5Y-D
			¼ BY 3-IN. PLAIN STEEL FLATS				
Hot-dip galvanized	2.49	0	TrY	5Y	2R-5Y	TrR-10Y	5R-25Y
Hot-dip aluminum	0.90	0D	0	0	0	0D	TrY-D
Lead coated	...	5R-75R*	3R-90R*	TrR-90R*	50R*	TrR-45R*	10R-75R*
			¼ BY 3-IN. COPPER-BEARING STEEL FLATS				
Electrogalvanized	0.66	50R	50R	35R-60Y	...	...	...
Hot-dip galvanized	2.67	5Y	TrY	5Y	1R-2Y	5R-20Y	5R-15Y
Hot-dip aluminum	0.66	0D	0	0	0	0D	TrY-D
Lead coated	...	70R*	2R-75R*	TrR-85R*	50R*	5R-45R*	10R-75R*
			NO. 792 WINDOW FRAME SECTION				
Hot-dip galvanized	2.60	0	TrY	TrY	TrR-10Y	3R-15Y	5R-20Y
Hot-dip aluminum	1.39	0	0	0	0	0D	0D

REPORT OF COMMITTEE A-7

ON

MALLEABLE-IRON CASTINGS[1]

Committee A-7 on Malleable Iron Castings held two meetings during the past year; at Atlantic City, N. J., on June 28, 1949, and in Chicago, Ill., on April 27, 1950. Subcommittee meetings were also held in Pittsburgh, Pa., on March 1, 1950.

During the year the National Electrical Manufacturers' Assoc. and the Maumee Malleable Castings Co. have discontinued their membership in Committee A-7 and D. I. Brown has been substituted for T. W. Lippert as the representative of *Iron Age*.

At the present time the committee consists of 57 members, of whom one is classified as "not voting"; of the 56 voting members, 28 are classified as producers, 14 as consumers and 14 as general interest members.

Committee A-7 is continuing its general interest in the project of working out some arrangement with the National Bureau of Standards by which a white iron sample will be added to the Bureau's list of standard samples.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. A. Kennedy.
Vice-Chairman, C. F. Lauenstein.
Secretary, James H. Lansing.

ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Pearlitic Malleable Iron Castings (**A 220 - 48 T**).[2]—These specifications have been in the process of development over a number of years and represent the consensus of all concerned in the field of production and use of pearlitic malleable iron. Some revisions, including the addition of a new grade 53004, were approved by Subcommittee IV at its meeting on March 1, 1950, and later received the approval of Committee A-7 on April 27, 1950.

The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard, with the following revisions:

Section 3 (*a*).—Revise to read "The tension test specimen made according to Section 4 shall conform to the minimum requirements as to tensile properties prescribed in Table I."

Revise the present tabular material to read as shown in the accompanying Table I.

TABLE I.—TENSILE REQUIREMENTS.[a]

	Grade 43010	Grade 48005	Grade 53004	Grade 60003	Grade 70002
Tensile Strength, min., psi.[b]	60 000	70 000	80 000	80 000	90 000
Yield Point or Yield Strength, min., psi.[b]	43 000	48 000	53 000	60 000	70 000
Elongation in 2 in., min., per cent	10	5	4	3	2

[a] Typical Brinell hardness number ranges of the grades of material in this table are as follows:

Grade	Typical Brinell Hardness Number Range
43010	163 to 207
48005	179 to 228
53004	197 to 241
60003	197 to 241
70002	241 to 285

[b] It is recognized that higher tensile and higher yield strengths may be obtained from any of the grades of material in the table by the use of a conventional quench-and-temper treatment.

Section 9.—Revise to read "All castings shall be substantially free from massive (primary) cementite, primary graphite, shrinks, cold shuts, cracks, injurious porosity, or other defects making the castings unsuitable."

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 1.

SPECIFICATION CONTINUED AS TENTATIVE

Tentative Specifications for Malleable Iron Flanges, Pipe Fittings and Valve Parts (**A 277 - 44 T**).[2]—The wording of these specifications, specifically Section 3 (*b*), has been the subject of discussion for a number of years, both when the specifications were published as Emergency Specifications ES-20 from 1942 to 1944 and subsequently, as they have stood unchanged during the past six years, as A 277 - 44 T.

At the time of the Annual Meeting in 1947, a special subcommittee (George E. Bean, chairman) was set up to study these specifications with the thought of resolving the differences in ideas, if possible. Although the recommendation made by this subcommittee, based on a report dated April 11, 1949, was that the tentative specifications be adopted as standard and this recommendation received a favorable majority of 23 for and 10 against its support at the time of the Annual Meeting of June, 1949, it was decided to refer the specification back to the subcommittee for further consideration during the current year.

By a vote of 6 for and 2 opposed, the subcommittee at its meeting of March 1, 1950, again took action to recommend Specifications A 277 - 44 T for adoption as standard without change in wording. This recommendation received the support of more than two-thirds of those who attended and voted at the meeting of Committee A-7 held in Chicago on April 27; at that meeting, 18 voted in favor, 7 were opposed and 4 did not vote.

A subsequent letter ballot of the entire committee was taken, 29 voting in favor of adoption, 10 opposing such action and 10 not voting. Of the producers, 18 were in favor, 8 were opposed, and 2 did not vote.

Although obtaining better than the required $\frac{2}{3}$ favorable vote for adoption, the committee at its meeting on June 26, 1950, deemed it wise to again defer action on this matter, leaving Specification A 277–44 T for another year without change, with the thought of making a renewed endeavor to work out something that will meet with the approval of all, or at least more nearly all, of the members.

To this end, the chairman has been authorized to appoint a permanent subcommittee with the present special subcommittee as a nucleus to be known as the Subcommittee on Malleable Iron Castings for Valves, Flanges, and Pipe Fittings.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Air Furnace, Electric, and Multiplex Malleable Iron (J. H. Lansing, chairman) has been giving consideration to two suggestions bearing on the wording of Specifications A 47 - 48 for Malleable Iron Castings. One has to do with a reported variation in wording between Specifications A 47 - 48 and the A.A.R. Specifications M 402 - 45 for Malleable Castings, with the possibility that an additional clause should be added to Specifications A 47. The other bears on the possible need for a provision on production control and maintenance of records.

Subcommittee IV on Pearlitic and Alloy Malleable Iron (C. F. Lauenstein, chairman) has completed consideration of Specifications A 220 - 48 T leading up to the recommendation that this specification be adopted as standard as explained previously in this report.

This report has been submitted to letter ballot of the committee, which consists of 57 members, one classified as "not voting" 36 members returned their ballots, of whom 33 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

W. A. KENNEDY,
Chairman.

J. H. LANSING,
Secretary.

REPORT OF COMMITTEE A-9
ON
FERRO-ALLOYS[1]

Committee A-9 on Ferro-Alloys held a meeting in Atlantic City, N. J., on June 29, 1949, and plans to meet there on June 28, 1950.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. C. Bowden, Jr.
Vice-Chairman, C. M. Loeb, Jr.
Secretary, G. F. Comstock.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1949 Annual Meeting, Committee A-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Ferroboron (A 323 - 49 T), and
Ferrotitanium (A 324 - 49 T).

Revision and Reversion to Tentative of Standard Specifications for:

Molybdenum Salts and Compounds (A 146 - 39).

These recommendations were accepted by the Standards Committee on August 19, 1949, and the new tentative specifications appear in the 1949 Book of A.S.T.M. Standards, Part 1.

Recommendations Affecting Standards

The committee is recommending revisions in three tentatives and seven standards, as itemized in the Appendix to this report. These specifications have been modified in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (E 29 - 49 T).[2]

These actions have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

Subcommittee Activities

Subcommittee I on Specifications (E. A. Lucas, chairman) recommended as editorial revisions various changes in the manner of expressing the chemical requirements of the seven standard specifications under the jurisdiction of Committee A-9. These revisions were in accordance with the rounding-off method of Recommended Practice E 29 and were approved unanimously by Committee A-9 letter ballot.

However, due to the extent of the revisions and some question as to whether all the revisions were simply editorial, it was decided to resubmit the specifications to letter ballot with revisions as outlined in the Appendix to this report. Also included are revisions in three tentative specifications to bring them in line with the standard specifications.

Subcommittee II on Sampling (W. C. Bowden, Jr., chairman) has met with representatives of Committee E-3 on

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 1.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Chemical Analysis of Metals, Division S, in order to discuss sampling procedures for ferroboron and ferrotitanium. These procedures will be included in the Standard Methods of Sampling Ferro-Alloys (E 32 – 42).[4] These methods now include procedures for sampling low-carbon ferrotitanium.

This report has been submitted to letter ballot of the committee, which consists of 29 members; 24 members returned their ballots, of whom 19 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. C. BOWDEN, JR.,
Chairman.

G. F. COMSTOCK,
Secretary.

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[4] 1950 Book of A.S.T.M. Methods of Chemical Analysis of Metals.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON FERRO-ALLOYS

In this Appendix recommendations are given affecting certain specifications, both standard and tentative, covering various ferro-alloys. These specifications appear in their present form in the 1949 Book of A.S.T.M. Standards, Part 1.

REVISIONS OF TENTATIVES

Specifications for Molybdenum Salts and Compounds (A 146 – 49 T):

Section 1.—Revise to read "These specifications cover standard grades of molybdenum salts and compounds."

New Section.—Add a new Section 2 to read as follows, renumbering subsequent sections accordingly:

> 2. *Basis of Purchase* (*a*) The material shall be furnished in the form of molybdenum oxide, molybdic oxide briquets, and calcium molybdate.
>
> (*b*) The reported percentage of molybdenum as obtained by chemical analysis shall be used to determine the billing of the material to the purchaser.

Table I.—Add Footnotes *a* and *b*. Footnote *a*, referenced after the title of the table, shall read "For purposes of determining compliance with those specifications, the reported analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]"

Footnote *b*, referenced after "Molybdenum, per cent" shall read "For purposes of determining the molybdenum content of any shipment, molybdenum shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*."

Specifications for Ferroboron (A 323 – 49 T):

Section 2.—Letter present section as Paragraph (*a*) and add a new Paragraph (*b*) to read "(*b*) The reported percentage of boron as obtained by chemical analysis shall be used to determine the billing of the material to the purchaser."

Table I.—Add Footnotes *a* and *b* as indicated above in Table I of Specifications A 146, Footnote *b*, referenced after "Boron, per cent," shall read "For purposes of determining the boron content of any shipment, boron shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*."

Specifications for Ferrotitanium (A 324 – 49 T):

Section 2.—Revise as indicated above for Section 2 in Specifications A 323, except that in Paragraph (*b*) use the word "titanium" in place of the word "boron."

Table I.—Add Footnotes *a* and *b* as indicated above in Table I of Specifications A 146. Footnote *b*, referenced after "Titanium, per cent," shall read "For purposes of determining the titanium content of any shipment, titanium shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*."

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[1] 1949 Book of A.S.T.M. Standards, Part 1.

TABLE I.—CHEMICAL REQUIREMENTS.[a]

	Grade A	Grade B	Grade C
Manganese, per cent[b]	16.0 to 19.0	19.0 to 21.0	25.0 to 28.0
Carbon, max., per cent	6.5	6.5	6.5
Phosphorus, max., per cent	0.25	0.25	0.25
Sulfur, max., per cent	0.05	0.05	0.05
Silicon, per cent	[c]	[c]	[c]

[a] For purposes of determining compliance to these specifications, the reported from analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]

[b] For purposes of determining the manganese content of any shipment, manganese shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*.

[c] May be obtained containing silicon as follows: 1.0 per cent, max., 1.0 to 2.0 per cent: 2.0 to 3.0 per cent; and 3.5 to 4.5 per cent.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Specifications for Spiegeleisen (A 98 - 41):

Section 2.—Letter present section as Paragraph (*a*) and add a new Paragraph (*b*) to read "(*b*) The reported percentage of manganese as obtained by chemical analysis shall be used to determine the billing of the material to the purchaser."

Section 3 (*a*).—Revise to read "The material shall conform to the requirements as to chemical composition prescribed in Table I."

Revise the chemical requirements now in this section to read as shown in the accompanying Table I.

Specifications for Ferromanganese (A 99 - 39):

Section 2.—Revise as indicated above

TABLE II.—CHEMICAL REQUIREMENTS.[a]

	Standard	Medium Carbon			Low Carbon	
		Grade A	Grade B	Grade C	Grade A	Grade B
Manganese, per cent.[b]	78.0 to 82.0	80.0 to 85.0	80.0 to 85.0	80.0 to 85.0	80.0 to 85.0	80.0 to 85.0
Carbon, max., per cent	7.5	as specified[c]	as specified[c]	as specified[c]	as specified[d]	0.75
Phosphorus, max., per cent	0.35	0.35	0.35	0.35	0.35	0.35
Sulfur, max., per cent	0.05	0.05	0.05	0.05	0.05	0.05
Silicon, max., per cent	1.2	1.2	1.5	2.5	1.2	7.0

[a] For purposes of determining compliance to these specifications, the reported analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]

[b] For purposes of determining the manganese content of any shipment, manganese shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*.

[c] Medium-carbon material may be obtained with the following maximum percentages of carbon: 3.0, 2.0, 1.5 and 1.0.

[d] Grade A low-carbon material may be obtained with the following maximum percentages of carbon: 0.75, 0.50, 0.30, 0.15, and 0.10.

TABLE III.—CHEMICAL REQUIREMENTS.[a]

	Grade A	Grade B	Grade C	Grade D	Grade E	Grade F	Grade G
Silicon, per cent[b]	90.0 to 95.0	80.0 to 90.0	72.0 to 79.0	47.0 to 52.0	25.0 to 30.0	14.0 to 18.0	8.0 to 14.0
Carbon, max., per cent	0.15	0.15	0.15	0.15	0.50	1.5	1.5
Sulfur, max., per cent	0.04	0.04	0.04	0.04	0.04	0.06	0.06
Phosphorus, max., per cent	0.05	0.05	0.05	0.05	0.06	0.15	0.15

[a] For purposes of determining compliance to these specifications, the reported analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]

[b] For purposes of determining the silicon content of any shipment, silicon shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*.

for Section 2 in Specifications A 98.

Table I.—Revise to read as shown in the accompanying Table II.

Specifications for Ferrosilicon (A 100 – 39):

Section 2.—Add a new Paragraph (*c*) to read as indicated above for Section 2(*b*) in Specifications A 98, using the word "silicon" in place of the word "manganese."

Table I.—Revise to read as shown in the accompanying Table III.

TABLE IV.—CHEMICAL REQUIREMENTS.[a]

	High Carbon	Low Carbon
Chromium, per cent[b]	60.0 to 75.0	65.0 to 75.0
Carbon, per cent	4.0 to 8.0	2.0 max.[c]
Silicon, max., per cent	3.0	1.5

[a] For purposes of determining compliance to these specifications, the reported analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]

[b] For purposes of determining the chromium content of any shipment, chromium shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*.

[c] May be obtained with the following maximum percentages of carbon: 2.0, 1.0, 0.50, 0.20, 0.15, 0.10, 0.06, and 0.04.

Specifications for Ferrochromium (A 101 – 42):

Section 2.—Add a new Paragraph (*c*) to read as indicated above for Section 2(*b*) in Specifications A 98, using the word "chromium" in place of the word "manganese."

Section 3 (*a*).—Revise as indicated above for Section 3 (*a*) in Specifications A 98, with a new Table I to read as shown in the accompanying Table IV.

Specifications for Ferrovanadium (A 102 – 39):

Section 2.—Revise as indicated above for Section 2 in Specifications A 98, except that in Paragraph (*b*) use the word "vanadium" in place of the word "manganese."

Section 3 (*a*).—Revise as indicated above for Section 3 (*a*) in Specifications A 98, with a new Table I to read as shown in the accompanying Table V.

Specifications for Ferromolybdenum (A 132 – 39):

Section 2.—Revise as indicated above for Section 2 in Specifications A 98, except that in Paragraph (*b*) use the word "molybdenum" in place of the word "manganese."

TABLE V.—CHEMICAL REQUIREMENTS.[a]

	Grade A	Grade B	Grade C
Vanadium, per cent[b]	30.0 to 40.0	35.0 to 45.0	35.0 to 45.0
Carbon, max., per cent	3.5	0.50	0.20
Phosphorus, max., per cent	0.25	0.10	0.10
Sulfur, max., per cent	0.40	0.20	0.10
Silicon, max., per cent	13.0	3.5	1.2
Aluminum, max., per cent	1.5	1.5	1.2

[a] For purposes of determining compliance to these specifications, the reported analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]

[b] For purposes of determining the vanadium content of any shipment, vanadium shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*.

Section 3 (*a*).—Revise as indicated above for Section 3 (*a*) in Specifications A 98, with a new Table I to read as shown in the accompanying Table VI.

Specifications for Ferrotungsten (A 144 – 38):

Section 2.—Revise as indicated above for Section 2 in Specifications A 98, except that in Paragraph (*b*) use the word "tungsten" in place of the word "manganese."

Section 3 (*a*).—Add a Footnote *a* after the list of chemical requirements, referenced after the words "chemical composition," to read the same as Footnote *a* in the new Table I of Specifications A 98.

TABLE VI.—CHEMICAL REQUIREMENTS.[a]

	Grade A	Grade B
Molybdenum, per cent[b]......	55.0 to 70.0	55.0 to 70.0
Carbon, max., per cent......	2.5	0.25
Phosphorus, max., per cent..	0.10	0.10
Sulfur, max., per cent.......	0.25	0.25
Silicon, max., per cent.......	1.5	1.5
Copper, max., per cent......	1.0	1.0

[a] For purposes of determining compliance to these specifications, the reported analysis shall be rounded off to the nearest unit in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (A.S.T.M. Designation: E 29).[1]

[b] For purposes of determining the molybdenum content of any shipment, molybdenum shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*.

Add a Footnote (*b*) referenced after "Tungsten, per cent" to read "For purposes of determining the tungsten content of any shipment, tungsten shall be reported to the nearest 0.01 per cent, applying the same rounding-off procedure as prescribed in Footnote *a*."

Revise the required tungsten content range from the present "70.00 to 80.00" to read "70.0 to 80.0" per cent, as specified. Revise the maximum silicon content from the present "1.00" to read "1.0" per cent.

REPORT OF COMMITTEE A-10

ON

IRON-CHROMIUM, IRON-CHROMIUM-NICKEL AND RELATED ALLOYS[1]

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys held meetings at Atlantic City, N. J., on June 29, 1949, during the Annual Meeting of the Society, and on March 2, 1950, at Pittsburgh, Pa.

The committee expresses its regret at the demise of W. G. Conant, alternate representative of the General Electric Co.

R. H. Heyer has replaced T. F. Olt as Committee A-10's representative on Committee E-1, Subcommittees II, III, IV and VII.

The present committee consists of 88 members, of whom 42 are classified as producers, 33 as consumers, and 13 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Jerome Strauss.
Vice-Chairman, L. L. Wyman.
Secretary, M. A. Cordovi.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 296 – 49 T), and

Heat-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (A 297 – 49 T).

These recommendations were accepted by the Standards Committee on December 21, 1949, and the revised tentative specifications appear in the 1949 Book of A.S.T.M. Standards, Part 1.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision of the following standard and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (**A 270 – 47**):

Section 8 (*b*).—Change to read as follows:

(*b*) The wall thickness at any point shall not vary more than 12½ per cent, over and under, from the average wall specified.

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Classification of Data (F. P. Peters, chairman).—Since the last Annual Meeting this subcommittee has completed its editorial work on the compilation of Data on Wrought and Cast Heat- and Corrosion-Resisting Steels and forwarded it to Society Headquarters for publication. The compilation of data has been published and is available as *Special Technical Publication No. 52a*.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Methods of Corrosion Testing (F. L. LaQue, chairman).—The most important activity of subcommittee IV during the past year was the Symposium on Evaluation Tests for Stainless Steels held at the Annual Meeting of the Society in June, 1949, in Atlantic City. This Symposium included ten papers and attracted as discussers several other outstanding authorities on the properties of stainless steels. The combined papers and discussions are available as a special publication of the Society[3] which represents an important addition to our knowledge of these alloys.

As an outgrowth of this Symposium, studies are under way to standardize further certain details of the acid copper sulfate test and the nitric-hydrofluoric acid test. In addition, data are being accumulated looking to the revision of the Tentative Recommended Practice for Boiling Nitric Acid Test for Corrosion-Resisting Steels (A 262 - 44 T) so as to permit the use of apparatus for exposing a large number of specimens in a single container, as described in the Symposium paper by DeLong.[3]

A special sub-group was organized under the chairmanship of E. G. Holmberg to survey methods for the cleaning of vessels to eliminate surface contamination or foreign metal and scale contamination, etc. after fabrication. This group will also explore suitable methods for inspection after such cleaning treatments.

Subcommittee IV is cooperating with a special committee of the High Alloys Committee of the Welding Research Council under the chairmanship of H. M. Brown. This committee is arranging for the preparation and subsequent long-time plant-corrosion testing of a large number of welded specimens of several compositions of stainless steel given various heat treatments. The plant tests will be made in many environments and the results will be correlated with laboratory evaluation tests.

The long-planned atmospheric corrosion testing program is still active and just as soon as a few final details can be cleared the accumulation and preparation of specimens for exposure will be begun.

Subcommittee V on Mechanical Testing (R. H. Heyer, chairman).—The results of the subcommittee's investigations on the effect of speed of testing on tensile properties of austenitic stainless steels were published in the A.S.T.M. BULLETIN of May, 1949, with a discussion in the December, 1949, issue.

At the March 1, 1950, meeting of the subcommittee in Pittsburgh it was concluded that the elapsed-time method and the rate-of-strain method of controlling speed of testing have merit, and that additional data are now needed to establish speed limits for the austenitic grades. Both of these methods of control will be used in tests designed to give this information. These tests will be carried out under carefully controlled conditions by two manufacturers of testing equipment.

The data on yield strength determination which were obtained in the fourth round robin were discussed in detail at the March 1 meeting. Limitations in equipment and testing technique and differences in interpretation of data were evident. Further work on this phase of tension testing is included in the test program mentioned above, which is under the chairmanship of R. A. Lincoln.

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[3] Issued as a separate publication, *STP No. 93*, 1949.

Subcommittee VI on Metallography (Russell Franks, chairman).—The work of this subcommittee on the determination of etching procedures for identification of the sigma phase in certain stainless steels has made considerable progress during the past year.

This work is being done on 18-8 steels with and without molybdenum. There are three steels involved: one which represents the type 304 (18-8) steel without molybdenum; one in which the 18 per cent chromium steel contains about 9½ per cent nickel with 3½ per cent molybdenum; and one in which the 18 per cent chromium steel contains 13½ per cent nickel and about 3½ per cent molybdenum. The steels have been held at temperatures between 1200 and 1500 F. and tested for impact toughness after exposure periods extending up to 500 hr. The results of the impact tests and the metallography work done on the fractured samples has shown that sigma phase does develop in the molybdenum-bearing chromium-nickel steels and that when substantial quantities of this phase are present the steels lose toughness to a very great extent. A foolproof etching procedure has not been developed to identify the sigma phase (without resorting to X-ray diffraction testing), but it is anticipated that within the coming year such a procedure will be developed that can be successfully used on stainless steels of known compositions.

Subcommittee VIII on Specifications for Wrought Products (J. K. Findley, chairman).—R. B. Gunia, who has been acting as temporary chairman of this subcommittee, reported the subcommittee had under consideration certain changes in Specification A 276 which would include the addition of type 430-F grade. A surface hardness test is under consideration for use in connection with this specification, also.

Subcommittee IX on Specifications for Flat Products (H. A. Grove, chairman).—This subcommittee has under consideration the shape and size of tension test specimens for plate over ½ in. in thickness and is also considering certain changes in Specification A 240, requested by the A.S.M.E. Boiler Code Committee.

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman).—This subcommittee carried out some matters by correspondence in connection with Specifications A 296 and A 297 during the year, which resulted in revision of these as mentioned earlier, and also reviewed a TAPPI Specification E-400 s-43, covering chromium-nickel-iron castings for sulfite paper pulp processing, and made recommendations pertaining thereto the Technical Association of the Pulp and Paper Industry. The subcommittee has now under study the problem of specifications for high-alloy centrifugal pipe castings and has appointed a task group under the chairmanship of W. F. Hodges to study this matter.

Subcommittee XI on Specifications for Tubular Products (J. J. B. Rutherford, chairman)—This subcommittee, cooperating in a joint A-1—A-10 Subcommittee on Stainless Steel Tubing Specifications, has under consideration modification of the Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 - 48 T), which was unsatisfactory for use under the A.S.M.E. Boiler Construction Code. A ballot on the changes required to make this specification acceptable is under way in the subcommittee and will be submitted to Committee A-10 for approval when the subcommittee's recommendations are received.

This joint subcommittee has under consideration a change in the wall thickness requirements in A.S.T.M. Specification A 270 for sanitary tubing which will likely result in having an over and under tolerance rather than the present stipulation wherein the wall thickness at any point shall not be less than 87.5 per cent of the specified average wall thickness.

This report has been submitted to letter ballot of the committee, which consists of 87 members; 49 members returned their ballots, of whom 48 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

H. D. NEWELL,
Secretary.

REPORT OF COMMITTEE B-1

ON

WIRES FOR ELECTRICAL CONDUCTORS[1]

Committee B-1 on Wires for Electrical Conductors held two meetings during the year, both at the Society's Headquarters in Philadelphia: one on January 20, 1950, and one on April 12, 1950. Meetings of the Advisory Committee and of Subcommittees II, IV, and VII also were held during the year.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. H. Stout, Jr.
Vice-Chairman, W. R. Hibbard.
Secretary, A. A. Jones.

Election of members-at-large of the Advisory Committee resulted in the selection of the following: G. W. Acock, G. H. Harnden, W. F. Markley, J. L. Thomas, and J. R. Thompson.

The subcommittee organization remains unchanged, except that B. J. Sirois has been appointed chairman of Subcommittee IV, succeeding Mr. Stout who assumes the chairmanship of Committee B-1.

The committee reports, with deep regret, that during the year it lost the service of its able Headquarters' sponsor, Carter S. Cole, who died on November 17, 1949. The retirement in the fall of 1949 of J. J. Morrison deprived the committee of another member whose active participation in its work for more than twenty years will long be remembered. Mr. Morrison subsequently was elected an Honorary Member of Committee B-1.

The committee now consists of 74 members, of whom 36 are classified as producers, 27 as consumers, and 11 as general interest members.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1949 Annual Meeting, Committee B-1 presented to the Society through the Administrative Committee on Standards the recommendation that the Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) be published as tentative. This recommendation was accepted by the Standards Committee on December 22, 1949, and the new tentative specifications appear in the 1949 Book of A.S.T.M. Standards, Part 2, bearing the designation B 245 - 49 T.

Revisions of Tentatives

The committee recommends that the following five specifications be revised as indicated and continued as tentative:

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (**B 172 - 49 T**):[2]

Note 5.—Add the following to this note: "In calculations involving density it must be borne in mind that apparent density of coated wire is not constant but a variable function of wire diameter.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 2.

The smaller the diameter, the greater the percentage of coating present and hence the greater departure from the density of copper."

Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Concentric Stranded Members for Electrical Conductors (**B 173 – 49 T**):[2]

Note 4.—Revise as indicated above for Note 5 in Specifications B 172.

Tentative Specifications for Bunch-Stranded Copper Conductors for Electrical Conductors (**B 174 – 49 T**):[2]

Note 3.—Revise as indicated above for Note 5 in Specifications B 172.

Tentative Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (**B 230 – 49 T**):[2]

Section 2.—Revise to read as follows: "The aluminum wire shall be made from rod meeting all the requirements of the Standard Specifications for Rolled Aluminum Rods (EC Grade) for Electrical Purposes (A.S.T.M. Designation: B 233)."

Section 3.—Reletter the present as Paragraph (*b*) and add a new Paragraph (*a*) to read as follows:

3. (*a*) A sample for chemical analysis shall be taken to represent each 4000 lb. or less of material in the lot. Sampling of the finished wire for check analysis shall not be required if the manufacturer can show that the analysis made at a prior stage of manufacture conformed to the requirements prescribed in Section 4 of Specifications B 233.

Section 4(*d*).—Reletter as Paragraph (*a*) of new Section 6 described below.

New Section.—Add a new Section 6 titled "Retests," renumbering subsequent sections accordingly. Paragraph (*a*) of this new section shall consist of the present Paragraph (*d*) of Section 4. Paragraph (*b*) is to read as follows:

(*b*) If on testing a sample from any reel, coil or spool of wire the results do not conform to the requirements of Section 5, two additional samples shall be tested and the average of the three tests shall determine the acceptance or rejection of the reel, coil, or spool.

Tentative Specifications for Concentric-Lay-Stranded Aluminum Conductors, Steel-Reinforced (*ACSR*) (**B 232 – 48 T**):[2]

Section 2(*b*).—Revise to read as follows: "Before stranding, the zinc-coated steel wires used shall meet all the requirements of the Tentative Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (A.S.T.M. Designation: B 245)."

Section 3(*b*).—Revise to read "There shall be no joints of any kind made in the finished zinc-coated steel wires."

Section 6(*b*).—Revise to read "Tests for all properties of zinc-coated steel wires shall be made before stranding."

Section 7.—Change the title of Section 7 to read "Strength of Conductor" and revise Section 7 to read as follows:

7. (*a*) The ultimate tensile strength of the completed conductor shall be taken as the sum of the ultimate tensile strengths of the aluminum wires, calculated from their nominal diameter and the appropriate specified minimum average tensile strength given in Table I of the Tentative Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (A.S.T.M. Designation: B 230) plus the sum of the values for stress at 1 per cent extension under load for the zinc-coated steel wires, calculated from their nominal diameter and the appropriate specified minimum stress at 1 per cent extension given in Table I of the Tentative Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (A.S.T.M. Designation: B 245).

(*b*) Tests for ultimate tensile strength of the completed conductor are not required by these specifications but may be made if agreed upon by the manufacturer and the purchaser. For this test, compression or other suitable fittings shall be employed and the breaking strength of the conductor when so tested shall be at least 95 per cent of the rated conductor strength (Note 4).

Section 9(*c*).—Change the word "cable" to read "conductor."

TABLE I.—RECOMMENDED SHIPPING LENGTHS FOR STANDARD SIZES OF ALUMINUM CONDUCTOR, STEEL REINFORCED.

(For Information Only)

Conductor Size, cir. mils or A.w.g.	Stranding		Weight per 1000 ft. lb.	Nominal Length of Each Piece, ft.	Nominal Weight of Each Length, lb.
	Aluminum	Steel			
1 590 000	54 × 0.1716	19 × 0.1030	2044	2310	4722
1 510 500	54 × 0.1672	19 × 0.1004	1942	2435	4729
1 431 000	54 × 0.1628	19 × 0.0977	1840	2570	4729
1 351 500	54 × 0.1582	19 × 0.0949	1737	2720	4725
1 272 000	54 × 0.1535	19 × 0.0921	1635	2885	4717
1 192 500	54 × 0.1486	19 × 0.0892	1533	3080	4722
1 113 000	54 × 0.1436	19 × 0.0862	1431	3300	4722
1 033 500	54 × 0.1384	7 × 0.1384	1331	3855	5131
954 000	54 × 0.1329	7 × 0.1329	1229	4170	5125
900 000	54 × 0.1291	7 × 0.1291	1159	4425	5129
874 500	54 × 0.1273	7 × 0.1273	1126	4555	5129
795 000	30 × 0.1628	19 × 0.0977	1235	3855	4761
795 000	26 × 0.1749	7 × 0.1360	1094	3590	3927
795 000	54 × 0.1214	7 × 0.1214	1024	5010	5130
715 500	30 × 0.1544	19 × 0.0926	1111	4290	4766
715 500	26 × 0.1659	7 × 0.1290	985	3985	3925
715 500	54 × 0.1151	7 × 0.1151	921	5575	5135
666 600	54 × 0.1111	7 × 0.1111	858	5975	5127
636 000	30 × 0.1456	19 × 0.0874	988	4810	4752
636 000	26 × 0.1564	7 × 0.1216	875	4490	3929
636 000	54 × 0.1085	7 × 0.1085	819	6260	5127
605 000	30 × 0.1420	19 × 0.0852	940	5070	4766
605 000	26 × 0.1525	7 × 0.1186	833	4715	3928
605 000	54 × 0.1059	7 × 0.1059	779	6580	5126
556 500	30 × 0.1362	7 × 0.1362	872	3965	3457
556 500	26 × 0.1463	7 × 0.1138	766	5125	3926
477 000	30 × 0.1261	7 × 0.1261	747	4635	3462
477 000	26 × 0.1355	7 × 0.1054	657	5975	3924
397 500	30 × 0.1151	7 × 0.1151	623	5555	3461
397 500	26 × 0.1236	7 × 0.0961	547	7180	3927
336 400	30 × 0.1059	7 × 0.1059	527	6565	3460
336 400	26 × 0.1138	7 × 0.0885	463	8470	3922
300 000	26 × 0.1074	7 × 0.0835	413	4750	1962
266 800	26 × 0.1013	7 × 0.0788	367	5340	1960
266 800	6 × 0.2109	7 × 0.0703	342	4975	1701
0000	6 × 0.1878	1 × 0.1878	291.1	4285	1247
000	6 × 0.1672	1 × 0.1672	230.8	5400	1246
00	6 × 0.1490	1 × 0.1490	183.1	5100	934
0	6 × 0.1327	1 × 0.1327	145.2	6440	935
1	6 × 0.1182	1 × 0.1182	115.2	*5405	623
2	7 × 0.0974	1 × 0.1299	106.7	8950	955
2	6 × 0.1052	1 × 0.1052	91.3	*6825	623
3	6 × 0.0937	1 × 0.0937	72.4	*8620	624
4	7 × 0.0772	1 × 0.1029	67.0	*7145	479
4	6 × 0.0834	1 × 0.0834	57.4	*10870	624
5	6 × 0.0743	1 × 0.0743	45.5	*13700	623
6	6 × 0.0661	1 × 0.0661	36.1	*17240	622
HIGH-STRENGTH CONDUCTORS					
211 300	12 × 0.1327	7 × 0.1327	527.5	6270	3307
203 200	16 × 0.1127	19 × 0.0977	676.8	7675	5194
190 800	12 × 0.1261	7 × 0.1261	476.3	4630	2205
176 900	12 × 0.1214	7 × 0.1214	441.4	4995	2205
159 000	12 × 0.1151	7 × 0.1151	396.8	5555	2204
134 600	12 × 0.1059	7 × 0.1059	336.0	6565	2206
110 800	12 × 0.0961	7 × 0.0961	276.6	7175	1985
101 800	12 × 0.0921	7 × 0.0921	254.1	8680	2206
80 000	8 × 0.1000	1 × 0.1670	149.0	5415	807

* Normally shipped 2 lengths per reel.

Section 10.—Revise to read as follows:

10. The area of cross-section of the aluminum wires of a conductor shall be not less than 98 per cent of the area specified. The area of cross-section shall be considered to be the sum of the cross-sectional areas of the component aluminum wires at any section when measured perpendicularly to their individual axes.

New Notes.—Add new Notes 6 and 7 to read as follows:

NOTE 6.—For the convenience of the users of these specifications, Table IV [the accompanying Table I] has been prepared giving recommended shipping lengths for the standard sizes of ACSR referred to in Tables I and II, based on the recommended steel wire coil weights given in the Tentative Specifications for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (A.S.T.M. Designation: B 245). Owing to the variation in coil weights, etc., it is common practice to allow a permissible variation in length of plus and minus 5 per cent for sizes larger than No. 1 Awg. and a tolerance of plus and minus 10 per cent on sizes No. 1 Awg. and smaller. It is also common practice to allow an amount not exceeding 10 per cent of the total weight of any one order to be shipped in random lengths but no piece shorter than 50 per cent of the standard length.

NOTE 7.—Wires unlaid from conductors will manifestly have different physical properties than those of the wire when prepared for cabling, on account of the deformation brought about by laying and again straightening for test. If tests on the aluminum and galvanized steel wires are to be made after stranding the purchaser and the manufacturer at the time of placing the order should agree on the properties to be met.

New Table.—Add a new Table IV to read as shown in the accompanying Table I.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in the following five standards and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (**B 8 - 49**):[2]

Note 8.—Revise as indicated above for Note 5 in Tentative Specifications B 172.

Standard Specifications for Cored, Annular, Concentric-Lay-Stranded Copper Conductors (**B 226 - 49**):[2]

Note 6.—Revise as indicated above for for Note 5 in Tentative Specifications B 172.

Standard Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (**B 33 - 46**):[2]

New Section.—Add a new Section 13 titled "Density" to read: "For the purpose of calculating weights, cross-sections, etc., the density of the copper shall be taken as 8.89 g. per cu. cm. (0.32117 lb. per cu. in.) at 20 C. (68 F.) (Note 8)." Renumber subsequent sections accordingly.

TABLE II.—PERMISSIBLE VARIATIONS IN DIAMETER.

Nominal Diameter,[a] in.	Permissible Variations, in.
¼	+0.020 −0.010
5/16	±0.015
⅜	±0.015
7/16	±0.015
½	±0.015
9/16	±0.015
⅝	±0.015
¾	±0.015
⅞	±0.020
1	±0.020

[a] Rods whose nominal diameters lie between sizes listed above shall be subject to the permissible variation given for the next larger size.

New Note.—Add a new Note 8 to read as follows:

NOTE 8.—The value of density of copper is in accordance with the International Annealed Copper Standard. The corresponding value at 0 C. (32 F.) is 8.90 g. per cu. cm. (0.32150 lb. per cu. in.). In calculations involving density it must be borne in mind that the apparent density of coated wire is not a constant but a variable function of wire diameter. The smaller the diameter the greater the percentage of coating present and hence the greater departure from the density of copper.

Standard Specifications for Hot-Rolled Copper Rods for Electrical Purposes (**B 49 – 41**):[2]

Section 6.—Revise to read "The diameter of the rod at any point shall not vary from that specified by more than the amounts prescribed in Table I [the accompanying Table II]."

Standard Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (**B 189 – 48**):[2]

New Section.—Add a new Section 11 to read as indicated above for new Section 13 in Specifications B 33, renumbering subsequent sections accordingly.

New Note.—Add a new Note 4 to read as indicated above for new Note 8 in Specifications B 33.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and is surveying the need for a proposed compilation of Committee B-1 standards.

Subcommittee II on Methods of Test and Sampling Procedure (J. B. Dixon, chairman) has a task group engaged in revising Specification B 1 to include sampling procedures based on statistical methods, as developed by Committee E-11 on Quality Control of Materials.

Subcommittee IV on Conductors of Copper and Copper Alloys (H. H. Stout, Jr., chairman) has a task group engaged in the preparation of specifications for the conductors of segmental-type, single-conductor cable in sizes up to and including 4,000,000 cir. mils.

Another task group is conducting an investigation to determine optimum concentration of sodium polysulfide solution for use in the tinning test prescribed in Specifications B 33 and B 189.

The task group on revision of significant figures in all tables of wire sizes has been collaborating with the National Bureau of Standards to establish bases of calculation to be used in the forthcoming revision of *Circular No. 31.*

Another task group is working on the preparation of proposed specifications covering the American Wire Gage. Still another task group is engaged in preparing a specification for tinned hard-drawn and medium-hard-drawn copper wire.

Subcommittee V on Conductors of Ferrous Metals (L. H. Winkler, chairman) has just started the preparation of a proposed specification for steel core wire (for ACSR) having heavier than standard zinc coating.

Subcommittee VI on Composite Conductors of Copper and Steel (C. E. Ambelang, chairman) is inactive at present, having completed an extensive program last year.

Subcommittee VII on Conductors of Light Metals (P. V. Faragher, chairman) is collecting data on wire characteristics (employing proposed sampling procedures based on statistical methods) so that basic data will be available for future revision of these requirements.

This report has been submitted to letter ballot of the committee, which consists of 74 members; 52 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. H. FOOTE
Chairman.

A. A. JONES,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable: the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE B-2

ON

NON-FERROUS METALS AND ALLOYS[1]

During the past year Committee B-2 on Non-Ferrous Metals and Alloys has handled all its work by correspondence. A number of standards and tentatives have been revised, certain tentatives have been advanced to standards, and the standards and tentatives under the committee's jurisdiction have been reaffirmed.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Bruce W. Gonser.

Secretary, G. Howard LeFevre.

The Advisory Committee met on September 23, 1949, at Society Headquarters in Philadelphia, approved changes in the membership of Committee B-2 and its subcommittees, and accepted reports on the work of the subcommittees.

At this meeting the Advisory Committee discussed the scope of work of Committee B-2, in particular the work of writing specifications for what are termed High-Temperature Non-Ferrous Metals and Alloys (Jet Alloys), and in order to clarify the scope of Committee B-2 the following resolution was passed: "Committee B-2 is to be functional on all specifications dealing with non-ferrous metals and alloys not otherwise assigned to some other committee as of this date (September 23, 1949)."

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee B-2 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

Revision of Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet and Strip (B 127 - 41 T),
Nickel-Plate, Sheet and Strip (B 162 - 41 T),
Nickel-Chromium-Iron Alloy Plate, Sheet and Strip (B 168 - 41 T),
Nickel-Copper Alloy Rods and Bars (B 164 - 41 T),
Nickel Rods and Bars (B 160 - 41 T),
Nickel-Chromium-Iron Alloys Rods and Bars (B 166 - 41 T),
Nickel-Copper Alloy Cold-Drawn Pipe and Tubing (B 165 - 41 T),
Nickel Cold-Drawn Pipe and Tubing (B 161 - 41 T),
Nickel-Chromium-Iron Alloy Cold-Drawn Pipe and Tubing (B 167 - 41 T), and
Nickel, Nickel-Copper Alloy, and Nickel-Chromium-Iron Alloy Seamless Condenser Tubes and Ferrule Stock, (B 163 - 41 T).

These recommendations were accepted by the Standards Committee on December 14, 1949, and the revised tentative Specifications appear in the 1949 Book of A.S.T.M. Standards, Part 2.

TENTATIVE CONTINUED WITHOUT REVISION

Pending clarification of the market condition for the material covered by Tentative Specification for Fire-Refined Casting Copper (B 72 - 47 T), the committee recommends that this tentative specification be continued in its present status without revision.

REAFFIRMATION OF STANDARDS

The committee, without dissenting vote, reaffirmed the following specifications, which have been published for six years or longer without revision and which are to be continued as such:

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

Standard Specifications for:

Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 4 – 42),
Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 43),
Nickel (B 39 – 22),
Rolled Zinc (B 69 – 39),
Lead-Coated Copper Sheet (B 101 – 40), and
Electrolytic Cathode Copper (B 115 – 43).

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Refined Copper (William E. Milligan, chairman) reviewed the specifications under its jurisdiction and reaffirmed the Standard Specifications for: Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots and Ingot Bars (B 4 – 42) and Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 43).

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman) is further reviewing Specification B 6 – 49 for Slab Zinc (Spelter).

Subcommittee VI on Coated Metals (F. L. Scovill, Jr., acting chairman) is continuing its study of Standard Specification for Lead Coated Copper Sheet (B 101 – 40) with an aim to provide certain methods of test for the continuity of coating.

Subcommittee VII on Refined-Nickel and High-Nickel Alloys, Cast and Wrought (O. B. J. Fraser, chairman) revised the ten Tentative Standards for Nickel and High Nickel Alloys which are listed under "Recommendations Accepted by the Administrative Committee on Standards."

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—There has been no work before this subcommittee during the past year. This subcommittee is prepared to consider specifications for high-temperature non-ferrous metals and alloys (jet alloys).

This report has been submitted to letter ballot of the committee, which consists of 118 members; 85 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

G. HOWARD LEFEVRE,
Secretary.

REPORT OF COMMITTEE B-3

ON

CORROSION OF NON-FERROUS METALS AND ALLOYS[1]

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held a meeting in Pittsburgh, Pa., on February 27, 1950.

The committee consists of 105 members of whom 87 are voting members; 28 are classified as consumers, 38 as producers, and 21 as general interest members.

The election of officers for the ensuing two years resulted in the reelection of the following:

Chairman, K. G. Compton.
Vice-Chairman, W. H. Finkeldey.
Secretary, A. W. Tracy.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following three methods be continued as tentative without revision:

Tentative Methods of:

Total Immersion Corrosion Testing of Non-Ferrous Metals (B 185 – 43 T),
Salt Spray (Fog) Testing (B 117 – 49 T), and
Alternate Immersion Corrosion Test of Non-Ferrous Metals (B 192 – 44 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Testing (N. L. Foltz, chairman).—This subcommittee is continuing the study of the salt spray test and has a working group under the chairmanship of W. D. McMaster studying the acetic acid-salt spray test, with special attention to its use for chromium-plated zinc-base die castings. At a meeting of the subcommittee in February, T. P. May of the International Nickel Co., Inc. and A. L. Alexander of the Naval Research Laboratory reported the results of tests using different salt sprays, including natural and artificial sea water, in regard to their effect on metals and metallic and paint coatings. Papers covering the work reported are being sponsored by Committee B-3.[2]

Subcommittee VI on Atmospheric Corrosion (W. H. Finkeldey, chairman).—All test specimens have been removed from the La Jolla, Calif., test site because the test area was needed by the Oceanographic Institute. Plate specimens will be cleaned and weighed and tension specimens will be pulled, in accordance with previously used methods. This action closes out the use of the La Jolla test site.

Specimens of light metals for the atmospheric corrosion test, being jointly conducted by Subcommittee VI of B-3 and Committee B-7 on Light Metals and Alloys, will be placed on exposure this summer.

Subcommittee VII on Weather (F. L. LaQue, chairman).—The principal activity of Subcommittee VII has been the work of the subgroup (C. P. Larrabee and O. B. Ellis) in exposing calibration specimens of zinc and steel at several of the A.S.T.M. atmospheric exposure sites.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] Two papers "Spray Testing with Natural and Synthetic Sea Water. II. A Study of Organic Coatings" and "A Synthetic Sea Water and Its Corrosion Characteristics in Salt Spray Testing," by T. P. May and A. L. Alexander appear on pages 1160 and 1131 of this publication.

Results of exposure for one year at several sites have been reported. Additional specimens remain in test for removal over a period of years. A paper based on work to correlate certain weather factors with the atmospheric corrosion of zinc and iron at Middletown, Ohio, was presented by O. B. Ellis, at the June, 1949, meeting.[3]

A preliminary survey of instruments and methods for measuring various kinds of atmospheric pollution that might be expected to affect corrosion was presented by L. G. von Lossberg at the February, 1950, meeting.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (L. J. Gorman, chairman).—Part I of the subcommittee's program, consisting of disk-type specimens of two types of magnesium alloys coupled galvanically to other metals and alloys, has been assembled and placed on exposure as follows:

New York, N. Y. ...	August 15, 1949
Kure Beach, N. C. ...	November 2, 1949
Panama Canal Zone ...	February 10, 1950
State College, Pa. ...	May 10, 1950

Part II of the program, consisting of spool-type specimens, is going forward and it is planned to place these couples on exposure before the end of the year. The preparation of the plate-type specimens for Part III of the program will then be undertaken.

In accordance with the action taken at the February, 1950, meeting of the committee, all of the iron specimens coupled with stainless steel have been removed from the test racks at New York, N. Y., Kure Beach, N. C., Altoona, Pa., and State College, Pa. The couples containing mild steel having an abnormally low copper content had been on exposure approximately 9 yr. and the couples with mild steel having a normal copper content had been on test for approximately 5 yr. The steels in all these couples were severely corroded. The contact resistance between disks on all other couples was found to be satisfactory, and it is planned to allow these couples of stainless steels with aluminum alloys, copper, bronze, antimonial lead, zinc, and monel to remain on test for a total of 15 yr. The first set of these specimens was removed in 1946 after five years' exposure and reported in the 1948 *Proceedings*.[4]

This report has been submitted to letter ballot of the committee, which consists of 87 voting members; 68 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

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[3] "Effect of Weather on the Initial Corrosion Rate of Sheet Zinc," *Proceedings*, Vol. 49, p. 152 (1949).

[4] *Proceedings*, Vol. 48, p. 167 (1948).

REPORT OF COMMITTEE B-4

ON

ELECTRICAL HEATING, RESISTANCE, AND RELATED ALLOYS[1]

Committee B-4 on Electrical Heating, Resistance, and Related Alloys has held three meetings since the presentation of its previous report: in New York, N. Y., on June 7, 8, and 9, 1949; in New York, N. Y. on November 2 and 3, 1949; and in Philadelphia, Pa., on February 28, March 1 and 2, 1950.

The present membership of this committee totals 68 members, of whom 29 are classified as producers, 34 as consumers, and 5 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On June 24, 1949, the Administrative Committee on Standards accepted the recommendation of Committee B-4 that the Specifications for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices be published as tentative. The new specifications appear in the 1949 Book of A.S.T.M. Standards, Part 2, bearing the designation B 239 - 49 T.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following 13 tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Temper of Strip and Sheet Metals for Electronic Devices (Spring Back Method) (B 155 - 45 T),[2]

Density of Fine Wire and Ribbon for Electronic Devices (B 180 - 45 T),[2]

Strength of Welded Joints of Lead Wires for Electronic Devices and Lamps (B 203 - 45 T),[2]

Surface Flaws in Tungsten Seal Rod and Wire (B 204 - 45 T),[2]

Equivalent Yield Stress of Thermostat Metals (B 191 - 44 T),[2]

Effect of Controlled Atmosphere Upon Alloys in Electric Furnaces (B 181 - 43 T),[2]

Modulus of Elasticity of Thermostat Metals (Cantilever Beam Method) (B 223 - 48 T),[2]

Tentative Method of Testing:

Wire for Supports Used in Electronic Devices and Lamps (B 157 - 45 T),[2]

Fine Round and Flat Wire for Electronic Devices (B 219 - 47 T),[2]

Tentative Method for:

Measuring Residual Stress in Cylindrical Metal-to-Glass Seals (B 218 - 47 T),[2]

Tentative Specifications for:

Round Nickel Wire for Lamps and Electronic Devices (B 175 - 45 T),[2]

Chromium-Nickel-Iron Alloy Castings (25-12 Class) for High-Temperature Service (B 190 - 45 T),[2] and

Nickel-Chromium-Iron Alloy Castings (35-15 Class) for High-Temperature Service (B 207 - 46 T).[2]

PUBLISHED AS INFORMATION

Committee B-4 recommends that the following proposed method of test be published as information, as appended hereto.

Proposed Method of Test for:

Relative Thermionic Emissive Properties of Materials Used in Electron Tubes

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2a]

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 2.

[2a] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommitte I on Electrical Heating Materials (C. L. Raynor, chairman).—The main function of this subcommittee is with life testing of wire product for electrical heating elements leading to appropriate specifications. Since the Annual Meeting, this group has been concerned with revision of specification B 76, mainly in the light of test equipment and test conditions. This revision is still in progress. Prominent in the year's activity was the publication of the paper by A. deS. Brasunas and H. H. Uhlig, "Some Observations on the Accelerated A.S.T.M. Life Test for Electrical Heating Wires."[3] This group also reviewed a comprehensive summary of eleven years of testing on a single wire lot, pointing up the difference in seasonal results on test values thought largely due to humidity conditions. The design of life test equipment is receiving new and pointed consideration in the light of the above.

Subcommittee V on Wrought and Cast Alloys for High-Temperature Use (R. D. Van Nordstrand, chairman) is concerned with methods of test and specifications for high-temperature alloys in electric furnaces. Over the past year the work program has included consideration of a method of test for intermittently heating and cooling specimens automatically, in relation to oxidation, and investigation of Alloy Casting Institute tests and report of thermal fatigue and hot gas attack. The importance of silicon in alloy composition is judged important and beneficial to corrosion resistivity. The above characteristics are the basis of future work for specifications.

Subcommittee VII on Thermostat Materials (P. H. Brace, chairman) is concerned with methods of test and specifications for thermostat metals. At the present time, the problem of "hardness" testing is active and an extensive field test program is nearing completion which is hoped will lead to specification status in the near future. While two types of testers show generally good agreement, some variance in results as a function of decreasing load and with surface films and have somewhat delayed progress.

This group has a section working on a method for low expansion tests such as invar field testing. Effects of variances in composition are also under consideration. A survey of possible future application of Method B 95 with modification is also under consideration.

Subcommittee VIII on Metallic Materials for Radio Tubes and Incandescent Lamps (S. A. Standing, chairman) is concerned with materials for electronic tubes and lamps and supports the activities of four creative sections on cathode materials, tungsten wire, and metal cone material.

Section A on Cathode has continued extensive work on determination of quality of nickel-base metal for "emitter" purposes, improving chemical test methods, establishing better tube "prove in" practice and continues to establish an improved foundation by means of melt comparison, chemical, metallurgical, and physical test approaches. Prominent work of this group has been pubpublicized in the ASTM Bulletin.

Section C on Tungsten Wire developed and put forward a tentative Method for Sag Testing, which has been favorably voted upon by Committee B-4. This group continues active on the many other problems affecting very fine wire for typical electronic tube and lamp works.

Section E.—This section, influenced by the requirements of metal-to-glass seals in television picture tubes, has completed a specification for a suitable metal for this service and is active on another at this time.

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[3] ASTM Bulletin, No. 161, October, 1949, p. 31.

Subcommittee IX on Methods of Test for Alloys in Controlled Atmospheres (P. H. Brace, chairman).—The present work program continues on deterioration investigation of "green-rot" effects and behavior of alloys in atmospheres containing sulfur and moisture.

Subcommittee X on Contact Materials (F. E. Carter, chairman) is dealing with problems relating to the life test, physical properties, hardness, standardization of forms and sizes, and related subjects. Life test in relation to welding characteristic, and "surety of contact making" are currently being worked upon. A proposed Tentative Method of Test for Determining Hardness of Contact Materials, is nearing completion. Progress is reported on standardization of forms and sizes.

This report has been submitted to letter ballot of the committee, which consists of 68 members; 55 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

F. E. BASH,
Secretary.

APPENDIX

PROPOSED METHOD OF TEST FOR RELATIVE THERMIONIC EMISSIVE PROPERTIES OF MATERIALS USED IN ELECTRON TUBES[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test covers a procedure for determining the comparative merits of various radio tube materials based on their effect upon the electrical characteristics of a typical high vacuum tube (standard diode).

Outline of Method

2. Specifically, the standard diode is used as a means for determining the acceptability for production of melts of cathode nickel alloys, lots of cathode coating material, and heater insulation coating materials. In all tests the material or process to be tested is substituted for the standard material or process listed in Sections 5 to 13, inclusive. The listed materials and processes constitute the control, or standard for comparison. For effective use of the standard diode it is imperative that every processing step be controlled to a high degree. Wherever possible the test and control materials must be randomized and intermixed after marking with positive identification.

Symbols

3. The symbols used in this method are as follows:

I_s = emission at 40 v., d-c.,
$\underset{\vee}{I}_s$ = emission under low-field conditions,
$\hat{I}_s$ = emission under pulsed conditions,
I_f = heater current,
E_f = heater voltage,
E_B = plate supply voltage,
E_P = actual plate voltage,
R_L = load resistance in series with plate,
K_T = cathode nickel brightness temperature, in degrees Centigrade,
$\bar{X}$ = average of reported readings,
R = range of reported readings,
r = ratio of test lot averages to control averages,
FM = figure of merit, and
QR = quality ratio.

Test Lot

4. A test lot shall, unless specifically excepted, result in a minimum of ten finished tubes. Sufficient quantities of parts shall be prepared on all tests to insure this final lot size. All tests shall include a control lot assembled and processed at the same time as the lot under test.

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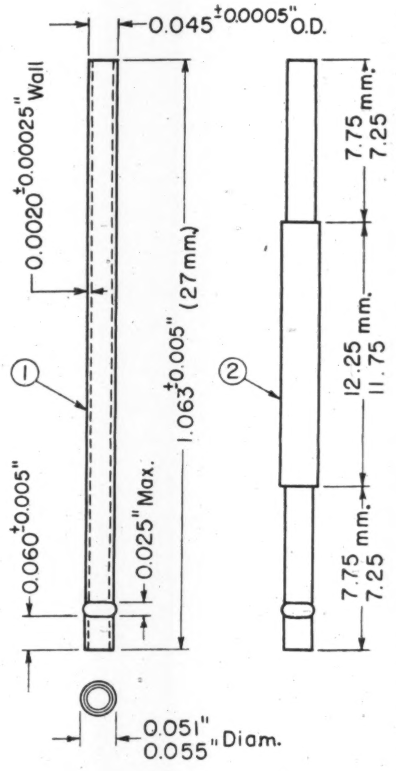
[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee B-4 on Electrical Heating, Resistance, and Related Alloys. Published as information, June, 1950.

Diode Tube Parts

5. (*a*) The standard diode shall have the following parts with the important dimensions listed (Detail drawings are shown in Supplement I.):



① Cathode sleeve

② 3.45 mg. triple carbonates

FIG. 1.—Cathode Assembly.

(*1*) *Cathode* (Fig. 1) (Note).—Nickel alloy sleeves made from tubing or strip with the following dimensions:

Outside diameter, in.	
seamless	0.045 ± 0.0005
lockseam	0.045 + 0.001 − 0.0005
Wall thickness, in	0.0020 ± 0.0025
Length, in	1.063 ± 0.005 (27.0 mm.)
Embossing location from one end, in	0.060 ± 0.005
Embossed diameter, in	0.053 ± 0.002
Embossed width, in	0.017 + 0.003 − 0.002
Connector tab ribbon, SiNi or Invar	0.015 in. by 0.005 in. by 11 mm.

NOTE.—For methods for physical testing of cathodes, see the Tentative Methods of Testing Sleeves and Tubing for Radio Tube Cathodes (A.S.T.M. Designation: B 128).[2] When cathode-base metals are being tested the control cathodes shall preferably be of the same alloy grade, see the Tentative Specifications for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (A.S.T.M. Designation: B 239).[2]

(*2*) *Plate.*—Grade 21 nickel strip is preferred conforming to the Tentative specifications for Circular Cross-Section Nickel Cathode Sleeves for Electronic Devices (A.S.T.M. Specifications: B 239):[2]

Strip thickness, in	0.005 ± 0.0005
Plate height, in	0.750 ± 0.010
Plate barrel inside diameter (round), in	0.132 ± 0.002
Plate width between center lines of two support glides, in	0.750 ± 0.004
Center line of main barrel	shall be within 0.0015 in. of common center line of support glides.

(*3*) *Mica Spacers.*—(two per mount), fair stain grade or better:

Thickness, in	0.010 ± 0.002
Cathode hole, in	0.0465 ± 0.0005
Plate support hole, in	0.0405 ± 0.0005
Center lines of holes	shall not deviate more than 0.001 in. from the design center line.

(*4*) *Bulb.*—T9-F1 lime glass or equivalent.

(*5*) *Stem.*—Lead glass to provide support for requisite wires and through connection leads.

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[2] 1949 Book of A.S.T.M. Standards, Part 2.

(*6*) *Connector Wires and Stay Ribbons.*—As required. Grade 21 of A.S.T.M. Specifications B 239 is preferred.

(*7*) *Heater.*—The heater shall conform to the following requirements:

Rating	6.3 v., 0.300 amp.
Shape	folded 6 legs; average of center legs, 27.5 mm. in length.
Wire, tungsten, mg. per 200 mm. of length	12 ± 0.2
Cut length, mm.	179 ± 1
Coated length, nominal, mm.	173
Diameter of coated material, in.	0.0075 to 0.0060
Coating material	alundum grain sintered to hardness. Hydrogen fired at 1500 C. or higher before tube assembly.

(*8*) *Getter.*—Barium or high barium alloy (free from magnesium). Inductive or resistance flashing.

(*b*) The cathode sleeves shall be spray coated with carbonate solution of the following composition (for detailed requirements see Sections A8 to A14 of Supplement I):

Carbonates	Barium, per cent by weight	57 ± 2
	Strontium, per cent by weight	39 ± 2
	Calcium, per cent by weight	4 ± 0.5
Binder	Nitrocellulose (25 sec.)	12.0 per cent
	Ethyl alcohol (dehydrated)	6.5 per cent
	Amyl acetate	81.5 per cent
Spray Mixture	Carbonates, g.	100
	Binder, ml.	30
	Amyl acetate, ml.	200
	Quantities shall be accurate to ± 3 per cent and shall be ground in a ball mill for 18 ± 4 hr.	

The mixture shall be applied with a spray gun to cathode sleeves held in a suitable spray bar to obtain a coating conforming to the following requirements:

Length of coated sleeve, mm.	12 ± 0.25 (7.5 mm. uncoated at each end)
Coated weight, mg.	3.45 ± 0.35
Diameter of coated sleeve nominal, in.	0.051
Area of coated cathode, sq. cm.	0.431
Apparent density of coating	0.8 mg. per cu. mm. ± 5 per cent
Unit weight of coating	8.0 mg. per sq. cm. ± 10 per cent

The coating diameter shall be measured with a barrel type micrometer reading to 0.0001 in. The diameter shall be measured by using the micrometer setting at which the cathode will just drop between the anvil and the spindle opening. In taking the measurement, the micrometer shall be set at a reading of less than cathode diameter, then adjusted until the cathode will just clear the opening by dropping through of its own weight. Coating thickness and density shall be controlled by taking samples at the middle and near each end of the spray bar, thus checking three cathodes from each spray bar. Before application, the guns and racks shall be thoroughly cleaned.

Parts Processing

6. All parts shall be cleaned and stored in accordance with best practices of the laboratory employing the diode test method. Detailed recommended schedules are given in Supplement I, Sections A3 to A6, inclusive. Several steps are recommended particularly as follows:

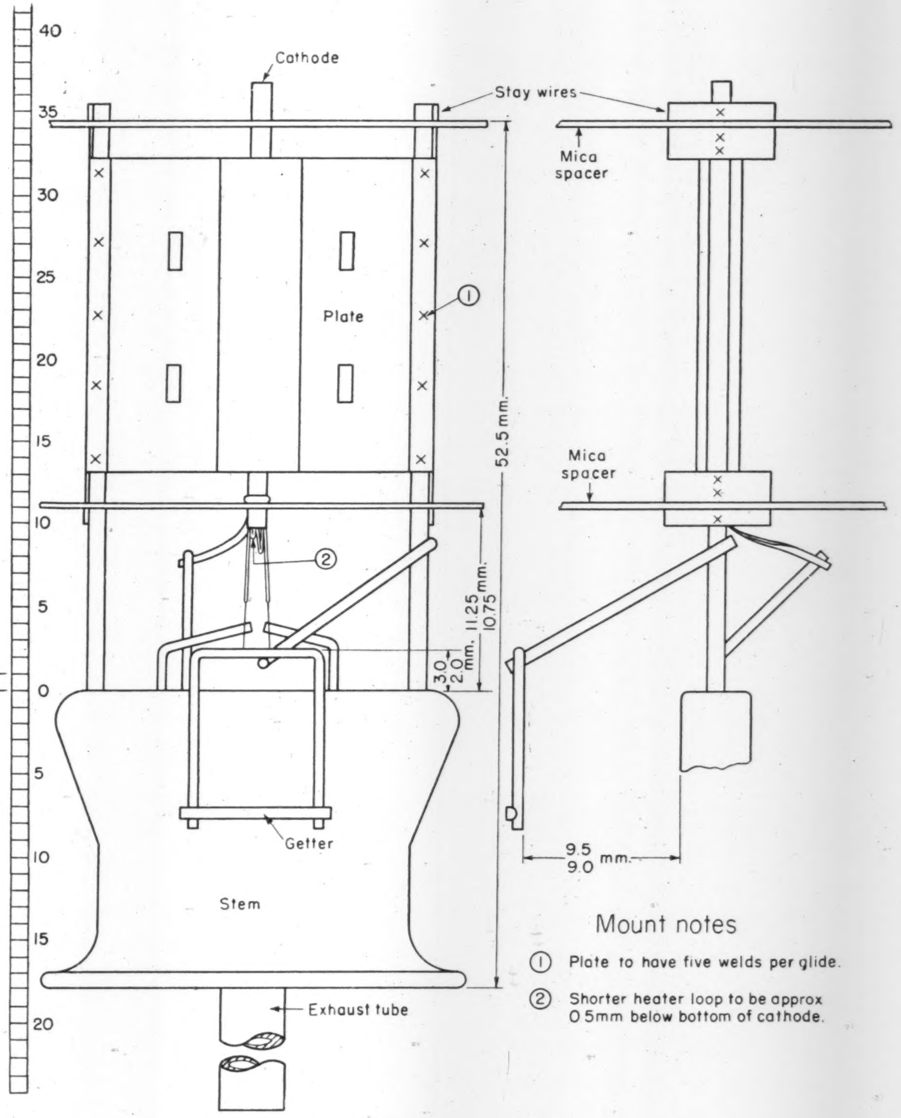
(*a*) Cathodes shall be fired in an atmosphere of dry hydrogen at temperatures between 575 and 600 C. for times between 5 and 10 min.

(*b*) Plates shall be selected to a uniform size for each group of comparative tests. The minor axis in the "as-stamped" condition (with no sizing) shall be within a range of plus or minus 0.0005 in.

(*c*) Mica shall be selected similarly to a thickness range within ±0.001 in.

Mount Assembly

7. (*a*) All parts shall be assembled into mounts (Fig. 2) in accordance with the best practices of the laboratory employing the diode test method. Oxide areas around welds or adherence of welder electrode material to the mount parts shall be avoided.


40
35
30
25
20
15
10
5
0
5
10
15
20
Scale — 1 mm.
Cathode
Stay wires
Mica spacer
Plate
①
②
52.5 mm.
Mica spacer
11.25 mm.
10.75
3.0 mm.
2.0
9.5 mm.
9.0
Getter
Stem
Exhaust tube

Mount notes

① Plate to have five welds per glide.

② Shorter heater loop to be approx 0.5mm below bottom of cathode.

FIG. 2.—Mount Assembly.

(*b*) Individual mounts and lots shall be identified by suitable scratched marks on the plate, or India ink marks on the top mica. All mounts from several lots being inter-compared shall be thoroughly mixed from this point until they are ready for electrical testing.

Sealing-In

8. Sealing-in may be carried out by either the manual or automatic process as described in the following Paragraphs (*a*) or (*b*), respectively:

(*a*) *Manual.*—When the manual process is followed the following items shall be observed:

(*1*) Sealing-in shall be carried out by best practices available. All mounts of several lots under comparative tests should be sealed at one time.

(*2*) In the event that sealed mounts will not be exhausted at once, they should be rough evacuated and tipped-off. Exhaust may then take place whenever feasible.

(*3*) During sealing-in the cathode temperature shall be kept below about 250 C. or shall be raised above about 350 C. If the cathode remains within this critical temperature range, the damage to the coating will be indicated by a caramel discoloration (see Section A14 in Supplement I). Also, the mount shall not reach a temperature which will cause the bright nickel parts to become oxidized.

(*b*) *Automatic.*—When the automatic process is followed the following items shall be observed:

(*1*) Sealing-in shall be done on an automatic machine which is currently producing good tubes of as near the same design as possible.

(*2*) Sealed mounts shall be transferred immediately to the exhaust. Mounts that are permitted to cool shall be discarded.

(*3*) On button stem construction, all fires shall be so directed as to present a minimum fire splash into the mount. All fires that have an opportunity of entering the mount shall be clean oxygen and gas fires.

(*4*) Conditions set forth in Paragraph (*a*), Item (*3*) are also applicable.

Exhaust

9. The exhaust procedure may be carried out by either the manual or automatic process as described in the following Paragraphs (*a*) or (*b*), respectively:

(*a*) *Manual.*—When the manual process is followed the following items shall be observed:

(*1*) Manual exhaust equipment shall be capable of pumping to less than 1 μ of mercury ultimate vacuum and have a pumping speed of greater than 25 l. per sec. at the tube.

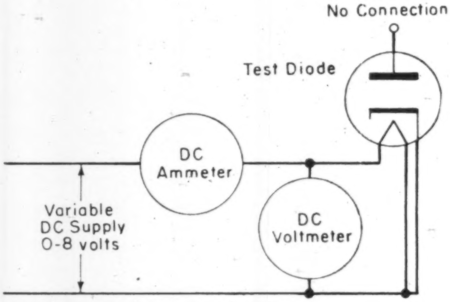


FIG. 3.—Circuit for Measuring Filament Current (I_f).

(*2*) Glass parts shall be baked to remove surface gas films.

(*3*) Metal parts shall be inductively heated to 800 ± 50 C. to remove adsorbed and absorbed gases.

(*4*) The cathode coating shall be broken down by an optimum schedule developed by each testing laboratory for each type of cathode material used for control purposes.

(*5*) Timing of each step of the exhaust procedure shall be accurate to within 1 sec.

(*b*) *Automatic.*—When the automatic

process is followed the following items shall be observed:

(*1*) The exhaust conditions should be normal to that of regular tube manufacture of a similar type.

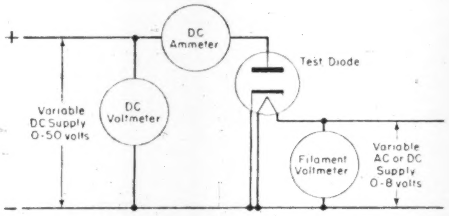

+
−
DC Ammeter
Test Diode
Variable DC Supply 0-50 volts
DC Voltmeter
Filament Voltmeter
Variable AC or DC Supply 0-8 volts

FIG. 4.—Circuit for Measuring Static Emission Currents (I_s) and ($\underline{I}_s$).

(*2*) Exhaust setup shall be capable of attaining a vacuum of 25 μ or less before reaching the getter flashing position. All port positions should pump to equal vacuum under equal load.

(*3*) The sealex shall be uniformly loaded, that is all ports full, or every other port, in order to insure uniform vacuum. Bombarding and breakdown lighting shall be controlled to eliminate any variations.

NOTE.—Suggested manual and automatic exhaust schedules are shown in Supplement I, Sections A15 to A18, inclusive.

Basing

10. Tubes shall be based prior to aging. Recommended basing arrangement (according to Radio and Television Manufacturers Association base pin number sequence) is as follows:

Pin Number	Element
2 and 7	heater
3	plate
8	cathode

NOTE.—To avoid arc-overs between lead wires or in the base and socket during pulse testing (where voltages are to exceed 2 kv.) it is recommended that the tubes be rebased so that the plate lead is connected to base pin No. 5.

Seasoning

11. Each tube shall be seasoned in accordance with optimum schedules as developed by each laboratory. Recommended schedules are shown in Sections A17 and A18 of Supplement I. The seasoning shall act to clean up residual gases, and bring the cathode to a high state of activation and stability. The emission, at $E_f = 6.5$ v. and $E_p = 40$ v., d-c. should be stable (at least on the control test) at the end of seasoning to the extent that it will not change by more than 3 per cent when drawn continuously for a minimum of 1 min.

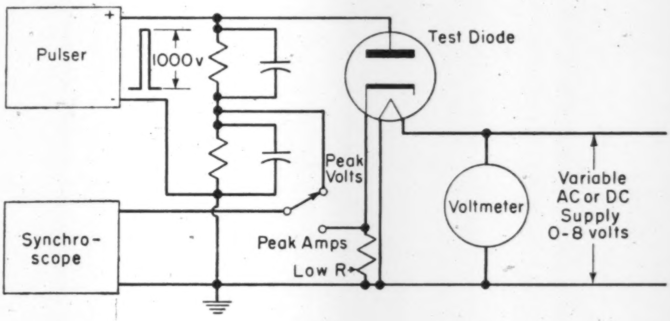


FIG. 5.—Circuit for Measuring Pulsed Emission Current ($\hat{I}_s$).

Testing

12. (*a*) Initial tests for diode emission shall be carried out under five conditions in the following sequence:

(1) Low Field ($\underline{I}_s$).
Preheat 1.75 v., E_f for 5 min.
$E_f = 1.75$ v. (a.c. or d.c.)
$E_p = 4.0$ v., d.c.
Report $\underline{I}_s$ in microamperes

(2) Pulse ($\hat{I}_s$).
- Preheat 4.5 v., E_f for 5 min.
- E_f = 4.5 v. (a.c. or d.c.)
- E_p = 1000 v. peak—square pulse
- Pulse width = 1 microsecond
- Pulse repetition rate = 200 pulses per second or less
- Report $\hat{I}_s$ in amperes, pulse shape, width and repetition rate

(3) 40 v., d-c. Emission (I_s).
- Preheat 7.0 v. E_f for 5 min.
- E_f = 7.0 v. to 3.0 v. (a.c. or d.c.) descending steps of 0.5 v.
- E_p = 40.0 v., d.c.
- Report I_s in milliamperes

NOTE.—At lower cathode temperatures, in the range of temperature-limited emission, slumping emission may occur. In such cases, peak instantaneous emission readings shall be taken. These readings shall be listed, followed by a downward arrow (that is, 35 ↓). Figures 3, 4, 5, and 6 for recommended testing circuits apply to the three conditions above.

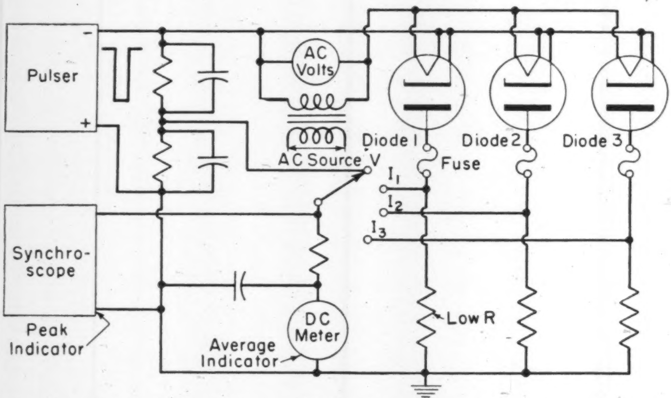
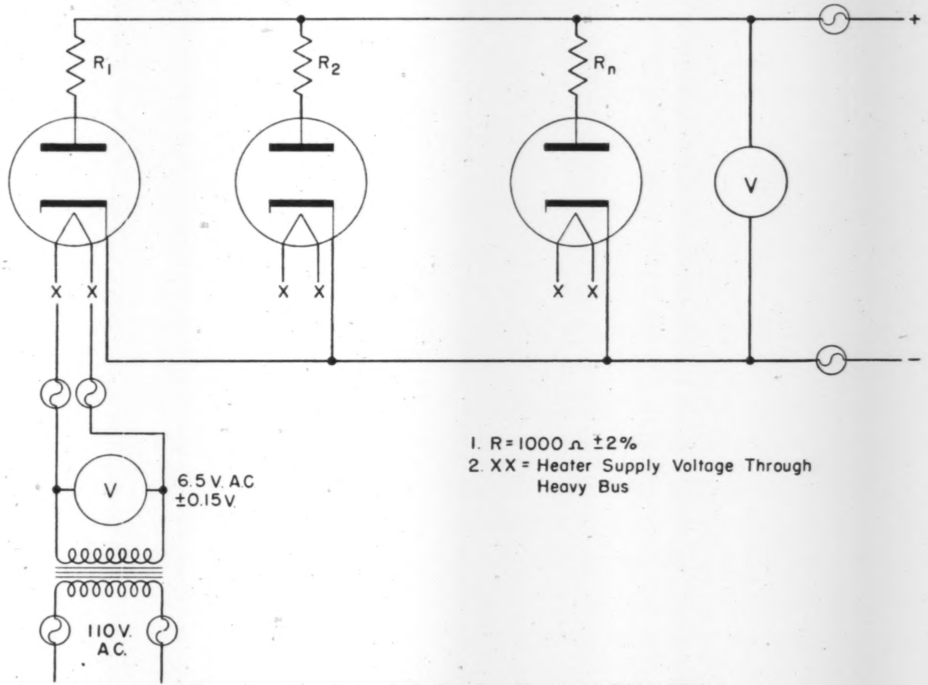


FIG. 6.—Circuit for Pulse Life Testing and Measuring Peak and Average Emission Currents.

(4) Filament Current (I_f).
- Preheat 6.5 v. for 1 min.
- E_f = 6.5 v. (d.c. recommended)
- Report I_f in milliamperes

(5) Cathode Temperature (K_t).
- Preheat E_f = 7.5 v. for 1 min.
- E_f = 7.5 v. (a.c. or d.c.)
- Report temperature of nickel cathode sleeve:
  - (*a*) Just above top of plate and below top mica spacer in degrees Centigrade, and/or
  - (*b*) As sighted through 0.040 in. diameter hole in center of plate and focusing upon the coating
- Record brightness temperature with optical pyrometer and location of temperature reading.

(*b*) It shall be standard practice to correct reported readings for meter currents or voltage drops where significant. Recommended practice for filament testing is to connect the filament voltmeter directly across tube terminals and to correct current readings for the filament voltmeter current. A direct current filament source and direct current meters are much to be preferred for most accurate and convenient filament current readings. Either alternating or direct current filament supply is suitable for all other tests. Recommended practice for plate current tests is to connect the d-c. voltmeter directly across the voltage source and to connect the d-c. ammeter between the source and the plate terminal of the tube. Correction here is made to the voltmeter reading due to voltage drop in the ammeter. This correction can generally be made insignificant through the use of ammeters having low voltage drop (of the order of one-tenth of a volt or less for full scale reading). All meters shall be periodically calibrated against meters having an accuracy of $\frac{1}{4}$ per cent of the full scale reading and readings shall be taken on the upper two-thirds of the scale wherever possible. Test equipment shall be calibrated in both heater and cathode-plate circuits by the use of stable precision resistors. The values of resistors shall be chosen to provide calibration readings in the ranges found for tube testing.



1. R = 1000 Ω ±2%
2. XX = Heater Supply Voltage Through Heavy Bus

FIG. 7.—Circuit for Life Testing Standard Diode Tubes.

Life Testing

13. (*a*) At least five tubes for each test lot and each control lot shall be life tested. Emission readings shall be taken at the intervals indicated. Life test conditions are as follows:

$E_f = 6.5 \pm 0.15$ v. d.c.
$E_B = 100 \pm 1.0$ v. d.c.
$R_L = 1000$ ohms $\pm$ 2 per cent.

This will provide an effective tube drop of about 40.0 v. and a cathode current of 60 ma. The plate dissipation will be about 2.4 w.

The schematic wiring diagram is shown in Fig. 7.

(*b*) Tubes shall be read for I_s (E_f = 6.5 v., 4.5 v., 3.5 v. and 3.0 v. with E_s = 40.0 v. d. c.) at the following time intervals: 0, 5, 25, 50, 100, 250 and 500 hr. (An alternative time schedule may be: 0, 5, 25, 50, 100, 200, 350, 500, 750, 1000, 1250, 1500 hr.) Readings should be taken within 5 per cent of the specified reading period. Tubes shall be allowed to remain without applied voltages at least 10 min. before making readings at down periods.

(*c*) Life tests should be discontinued when the 40 v. d-c. emission value at E_f = 4.5 v. has dropped to 50 per cent of the initial value of the control lot (this does not include early hour slumping, or low initial values which may be obtained with passive or slow activation materials).

(*d*) Pulse emission ($\hat{I}_s$) and low field emission ($\underset{\vee}{I}_s$) readings should be taken at the end of life. Additional readings are optional.

Report

14. Results of the test shall be reported as follows:

(*a*) Curves of d-c. emission (E_s = 40.0 v. d.c.) shall be presented, using rectangular coördinates. The test and control lots should be plotted on the same graph sheet.

(*b*) Test results on individual tubes which differ widely from the average of the lot, due to assignable causes, shall be omitted from consideration.

(*c*) At least two tubes of each lot shall be opened before the life test and two after the life test. Their dimensions and appearances shall be analyzed and reported in accordance with instructions in Supplement II.

(*d*) A precautionary test for gas shall be included. This test shall be made by measuring space-charged emission current at two different anode voltages. The test conditions are:

$$E_f = 6.5 \text{ v.} \quad E_b = 40 \text{ v. and } E_b = 20 \text{ v. d.c.}$$
$$\text{Read } I_s.$$

The ratio of I_s (40 v. E_b): I_s (20 v. E_b) should have a value of $2^{3/2} \equiv 2.8$. A significantly higher value than this, or higher than a normal value based on accumulated history, will indicate qualitatively the degree of gas in the tube.

(*e*) The preferred method of reporting results is as follows:

(*1*) The appended forms (Tables I and II) for reporting test results, processing and analytical data should be employed. It is recommended that at least ten good tubes be read for each test reported. With the average of control lot readings used as a base of 1.000, the ratio of the test to the control readings is to be calculated ("*r*" in Table I).

NOTE.—Control lots should show a reproducible I_s curve on successive runs. They should have a slope between E_f = 6.5 to 4.5 v. which is uniform and not to exceed 1.25 ma. per v. The slope should average 1.0 ma. per v. Absolute values of points on this section of the curve for successive control runs should lie within ±1 ma. Test lots having a slope of not more than 1.0 ma. per v. greater than 1.25 (or a total of 2.25 ma. per v.) are considered as not having significant difference from the control for production purposes.

(*2*) Life test results shall be reported on rectangular graph paper. The ordinate shall represent time in hours, the abscissa shall represent I_s in milliamperes. Indicate the average emission values at E_f = 4.5 and 6.5 v. for the test lots and control lots (Note) for each reading period.

(*f*) An alternate method of reporting *FM* values and other results is as follows:

(*1*) The report forms (Tables I and II) shall be used, with the exception that the range, *R*, of each set of readings shall replace the ratio of values, *r*, with respect to the control.

(*2*) The d-c. emission figure of merit *FM*, shall also be shown on the report form, as well as the quality ratio, *QR*. They are calculated as follows:

Plot log I_s *versus* log E_f on single cycle log-log graph paper. Use 10 ma. I_s and 1.0 v. E_f as coordinates at the origin. Draw a line from the origin which is tangent to the knee of the d-c. emission curve. Divide the I_s value at the point of tangency (Note) by the heater voltage for this same point. This is the *FM*. Then divide the figure of merit for the test lot by the figure of merit for the control lot. Multiply this value by 10 and round off to one decimal place. This is the *QR*.

NOTE: *Example*.—Point of tangency of curve and construction slope line = 40.3 ma. I_s ÷ 3.26 v. E_f = 12.36 (*FM* for control). 42.4 ma. I_s ÷ 5.74 v. E_f = 7.39 (*FM* for test lot). 7.39 ÷ 12.36 = 0.598 × 10 and rounding to one decimal place = 6.0. This figure is the d-c. emission quality ratio, *QR*. Values below 10 indicate that the test lots are poorer than the control. Values above 10 indicate the test lot to be better than the control.

(*3*) Life tests shall be reported by graphs, where the ordinate represents the time in hours, and the abscissa the values of *FM* or *QR*. When emphasis is desired upon early hour life performance, use semi-log graph paper (log on time axis up to 1000 hr.). Plot the test and control lots on the same graph sheet.

SUPPLEMENT I

DETAILED REQUIREMENTS AND SUGGESTED PROCESSING PROCEDURE FOR THE STANDARD DIODE

Parts

A1. The following sources of supply are suggested:

Part	*Suggested Supplier*	*Part Number*
Cathode	Superior Tube Co.	Seamless-S45R2-6 Lockseam L45R2-10
Heater (Fig. 8)	Sylvania Electric Product	F-109W1
Plate (Fig. 9)	Byer Manufacturing Co.	
Mica spacers (Fig. 10)	Ford Radio and Mica Co.	
Base	Owens-Illinois Glass Co. or Westinghouse Electric Co.	Octal Intermediate OCT6-46
Bulbs, T9-F1, lime	Corning Glass Works	
Stem (Fig. 11)	Raytheon Manufacturing Co.	
Spacer ribbon	Driver-Harris Co.	
Getter	Kemet Labs., Div. of National Carbon Co. R.C.A. Victor Division of R.C.A.	KIC 697 Batalum FZ323AC
Triple Carbonates	R.C.A. Victor Division of R.C.A.	33-C-130
Nitrocellulose Binder (Pyroxylin)	E. I. duPont de Nemours and Co., Inc.	5511

Cathode

A2. Material for the control normally used in the standard diode is International Nickel Co. 220 Alloy—a selected sample known as Melt No. 66. The chemical requirements are as prescribed in Table I.

If other base metal materials are being compared, one heat of that alloy is to be selected as a control for testing the other heats. The same is true for other materials subjected to the diode test.

RECOMMENDED PARTS CLEANING PROCEDURES FOR STANDARD DIODE

Processing of Metal Parts

A3. All metal parts shall be processed according to the following schedule:

(*a*) Wash in boiling trichlorethylene solution 5 min. using a wire mesh basket to hold the parts. The size of the baskets shall be such that the parts can be placed not more than three layers deep across the bottom of the tray. The trichlorethylene shall be stabilized for metal degreasing and shall have a residue content of not over 0.002 per cent on evaporation at 100 C.

(*b*) Rinse in trichlorethylene for 5 min.

(*c*) Dry for 5 min. at 100 C.

(*d*) Wash in boiling distilled water 5 min.

(*e*) Rinse in boiling distilled water 5 min. The distilled water shall have a residue content of not over 0.002 per cent determined by evaporation to dryness at 100 C. The conductivity of the distilled water shall be 300,000 ohms per cu. cm. or better. These provisions apply throughout this method whenever distilled water is specified.

(*f*) Rinse in acetone and air dry. The acetone shall be ACS reagent grade or better. This grade is also to be used throughout this specification.

(*g*) Oven dry using precautions to keep sample free from dust or other contamination.

(*h*) Fire at 600 C. for 10 min. in a hydrogen atmosphere that will bright anneal nickel-chromium or chromium-steel alloys. This is approximately equivalent to a dew point of −60 C.

Processing of Mica Parts

A4. Mica spacers shall be prepared by the following procedure:

(*a*) Rinse in distilled water for 5 min.

(*b*) Rinse in methanol and air dry. The methanol shall be ACS reagent grade or better. This provision applies to all methanol called for in this specification.

(*c*) Fire in hydrogen at 600 C. for 8 min. The hydrogen shall be at least 99.5 per cent pure and have a dew point of 0 C. or better.

Processing of Bulbs and Stems

A5. Bulbs and stems shall be cleaned as follows:

(*a*) Rinse in boiling distilled water for 3 min.

(*b*) Rinse in boiling distilled water for 1 min.

(*c*) Rinse in methanol and air dry.

(*d*) Dry for 5 min. at 150 C. in oven, using precautions to keep free from dust or other contamination.

Storage

A6. All parts and assemblies shall be stored in dustproof containers or wrappings in heated cabinets. The temperature of the cabinet shall be 40 ± 2C.

TABLE I.—CHEMICAL REQUIREMENTS FOR CATHODE MATERIAL.[a]

	Nickel plus Cobalt, per cent	Carbon, per cent	Copper, per cent	Iron, per cent	Manganese, per cent	Sulfur, per cent	Silicon, per cent	Magnesium, per cent
No. 220 Alloy	99.0	0.15	0.20	0.20	0.20	0.008	0.01 to 0.05	0.01 to 0.10
Melt No. 66[b,c]	99.7	0.06	0.02	0.07	0.10	0.004	0.03 max.	0.038

[a] Maximum per cent except where range is shown.

[b] Believed most reliable percentage composition from reports of several laboratories.

[c] Additional chemical analyses for Melt No. 66 are as follows:

Aluminum, per cent	0.003	Chromium, per cent	0.0008
Boron, per cent	0.003	Phosphorus, per cent	0.002
Cobalt, per cent	0.75	Titanium, per cent	0.03

Heater Coating

A7. The tungsten heater-wire shall be coated with a mixture of alundum powder and hydrogen fired at 1500 C. or above before use. It is suggested that the heater coating contain 1 part of 38–900 alundum to 3 parts of 38–500 alundum. The vehicle may be aluminum nitrate in water. The coated-wire diameter should be approximately 0.0075 to 0.0060 in. for a base wire size of 0.0014 in. It should not be brittle and subject to fracture upon normal handling and forming. The coating must be thoroughly cleaned from the legs of the heater at the points for welding to the stem wires.

RECOMMENDED PRACTICE FOR CATHODE COATING AND METHOD OF APPLICATION FOR THE STANDARD DIODE

Carbonate

A8. The carbonate shall be a co-precipitated triple carbonate containing 4 per cent calcium carbonate, the remainder consisting of approximately equal molecular portions of barium and strontium carbonate. The particle size shall average within the range of 1 to 9 μ with a maximum particle size of 25 μ. The chemical composition shall be as follows:

Barium carbonate, per cent	57.2 ± 2
Strontium carbonate, per cent	38.8 ± 2
Calcium carbonate, per cent	4.0 ± 0.5
Water soluble matter, per cent, max.	0.10
Iron, per cent, max.	0.003
Chloride, per cent, max.	0.003
Heavy metals, per cent, max.	0.003
(HCl) acid insoluble	0.01

The carbonate shall be dried for 12 hr. at 110 C. just before using and allowed to cool in a dry atmosphere.

Selection and Handling of Carbonate

A9. A lot of carbonate shall be selected in sufficient quantity to cover a 2-yr. minimum period. The carbonate shall conform to the requirements of Section A8 and shall be approved for use by the tube diode test using a previously approved lot of cathode base material. The carbonate shall be stored dry.

Binder for Cathode Carbonate

A10. (*a*) *Chemical Composition.*—The stock binder for the cathode carbonate shall consist of a solution of nitrocellulose in amyl acetate (du Pont No. 5511 pyroxylin) with the following chemical composition:

Nitrocellulose (25 sec.), per cent	12.2
Ethyl alcohol (dehydrated), per cent	6.6
Amyl acetate, per cent	81.2
Total solids, per cent	12.2

(*b*) *Viscosity.*—The viscosity of the binder shall be 36 to 50 sec. at 25 C. determined in accordance with the Standard Specifications and Tests for Soluble Nitrocellulose (A.S.T.M. Designation: D 301).[3]

(*c*) *Appearance.*—The binder shall be a clear, slightly yellow, syrupy liquid.

(*d*) *Packaging and Shipping.*—The material shall be shipped in airtight sealed 1-gal. cans.

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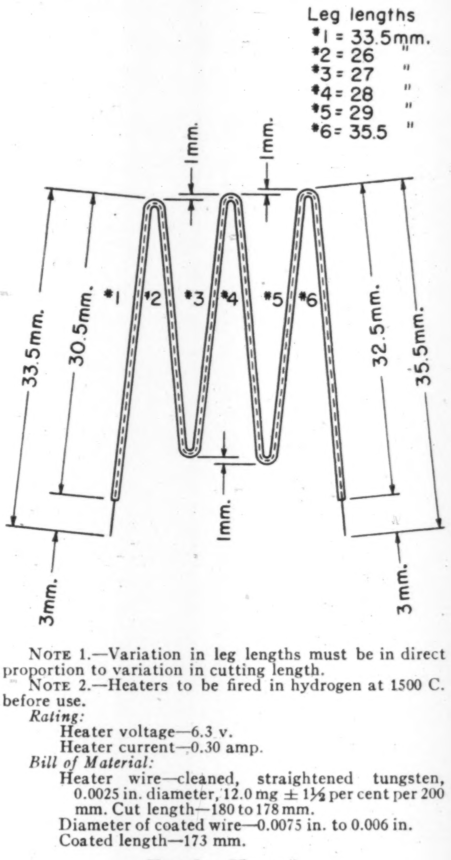
[3] 1949 Book of A.S.T.M. Standards, Part 4.

Spray Mixture

A11. (*a*) With each 200 g. of carbonates 60 ml. of stock binder solution diluted with 400 ml. of amyl acetate (see Paragraph (*c*)) shall be used. Quantities shall be weighed and measured to an accuracy of ±3 per cent. The viscosity of the binder shall be 16 ± 5 centipoises. The mixture shall be ground in a ball mill for 18 ± 4 hr. using a 1-qt. porcelain mill and porcelain balls. Suggested requirements for the ball mill shall be as follows:

Volume of mill, ml.	980
Volume of pebbles, ml.	235
Weight of pebbles, g.	600
Number of pebbles	80
Average weight of pebbles, g.	7.5
Weight of largest pebbles, g.	17.5
Weight of smallest pebbles, g.	3.5
Rotational speed of mill	70 rpm ± 10 per cent

(*b*) The viscosity of the milled spray mixture shall be 38 ± 1 centipoise at 25 C. The viscosity of the binder and of the spray mixture may be determined either with a Brookfield synchrolectric type viscometer or the Hercules capillary tube.



NOTE 1.—Variation in leg lengths must be in direct proportion to variation in cutting length.

NOTE 2.—Heaters to be fired in hydrogen at 1500 C. before use.

Rating:

Heater voltage—6.3 v.
Heater current—0.30 amp.

Bill of Material:

Heater wire—cleaned, straightened tungsten, 0.0025 in. diameter, 12.0 mg ± 1½ per cent per 200 mm. Cut length—180 to 178 mm.
Diameter of coated wire—0.0075 in. to 0.006 in.
Coated length—173 mm.

FIG. 8.—Heater.

(*c*) The amyl acetate used in the binder preparation shall conform either to the requirements of the Standard Specifications for Amyl Acetate (Synthetic) (85 to 88 per cent grade) (A.S.T.M. Designation: D 318),[3] or the Standard Specifications for Amyl Acetate Made from Fusel Oil (A.S.T.M. Designation: D 554).[3]

(*d*) The spray mixture may be packed and shipped in tinned cans or drums. Stoppers of the cans shall be metallic or foil-covered and shall be sealed.

Preparation of Cathode Sleeves

A12. For the preparation of cathode sleeves reference should be made to Section A3.

Coating Application

A13. (*a*) The coating shall be applied uniformly to the cathode sleeve with an air spray gun of the DeVilbiss CV type or its equivalent. The cathode shall be mounted and sprayed on a fixture holding 25 to 60 cathodes spaced sufficiently far apart to avoid turbulence. The test and control sleeves shall be randomized in the same rack. The texture of the coating shall be specified according to the diameter and weight in terms of apparent density. The apparent density of the coating shall be 0.8 ± 5 per cent with a coating weight of 8 mg. ± 10 per cent per sq. cm. The coating diameter shall be measured with a barrel type micrometer reading to 0.0001 in. The diameter shall be measured by using the micrometer setting at which the cathode will just drop between the anvil and the spindle opening. In taking the measurement, the micrometer shall be set at a reading of less than the cathode diameter, then adjusted by light tapping until the cathode will just clear the opening.

(*b*) The coating thickness and density shall be controlled by taking samples at the middle and near each end of the spray bar, thus checking 3 cathodes from each spray bar. Before application, the guns and racks shall be thoroughly cleaned.

Sealing-In

A14. (*a*) Sealing temperatures may be checked by visual examination of cooled mounts cut apart after sealing. Presence of a caramel discoloration trace on the cathode coating should subject results of tubes from that run to suspicion. The cathode coating may increase in temperature after tubes are removed from the sealing head due to heat conduction from hotter parts of the assembly.

(*b*) Proper sealing conditions may be readily checked by application of calibrated fusion point lacquers.[4] The indicating temperature range of the lacquer, the points of application, and the time and place of fusion require standardization by each laboratory. Cathode discoloration is the standard of comparison.

(*c*) Experience in the production of electron tubes confirmed by the diode investigations shows that emission is adversely affected when the coating binder is discolored during the sealing operation. It is believed that the nitrocellulose binder is partially decomposed and remains in a form which does not entirely leave the coating. Also, the darker color may affect the operating temperature. Not only is the emission particularly low at reduced sleeve temperatures, but it slumps—which is indicative of poisoning. Extreme accuracy of fire setting and control is necessary to avoid the critical temperature range. There does not seem to be appreciable difference for initial emission or its maintenance during life between tubes whose cathodes have been kept below or above the temperatures causing discolored coating.

Exhaust (*Automatic*)

A15. The following is a typical set up for automatic exhausting at an index speed of 800 positions per hour:

Position	Evacuation	Bombarding	Lighting Current, ma.
1....	Load sealed mount	...	...
2....	Port clamp opened	...	...
3....	Rough vacuum-leak detector	...	...
4....	Rough vacuum	...	...
5....	Fine vacuum pumping	Preheat	480
6....		Dull red	520
7....		Red	510
8....		Red	510
9....		Red	510
10....		Red	490
11....		Red	490
12....		...	475
13....		Getter flash	470
14....		...	455
15....	Tip off	...	...
16....	...	...	...

Exhaust (*Manual*)

A16. A suggested exhaust schedule for stationary exhaust equipment is as follows:

(*a*) Pumping system should include diffusion and forepumps capable of attaining an end

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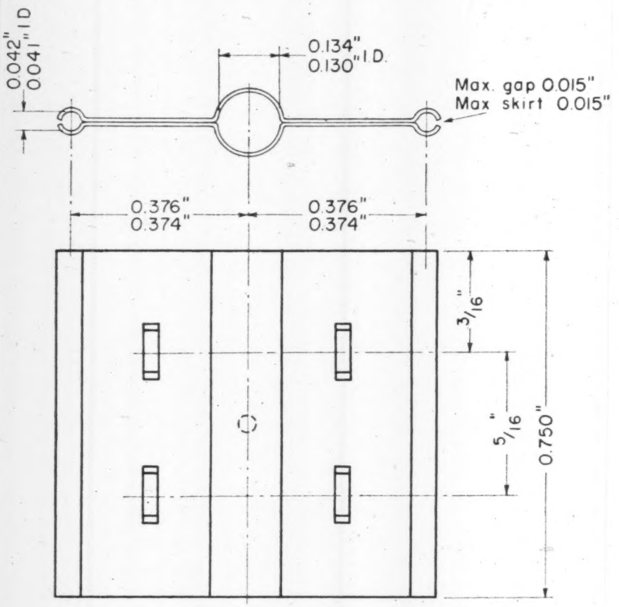
[4] Obtainable from Tempil Corp., New York 11, N. Y., under trade name of Tempilaq. Temperature indications are in 25 ± 2 F. steps in the desired sealing range.



NOTE.—Center line of main barrel to be within 0.0015 in. of common center line of glides. Hole stamped in half plate section, optional for optical cathode coating temperature measurements.
Plate—0.005 in. nickel, 0.750 in. wide.

FIG. 9.—Plates.

vacuum of less than one μ of mercury and with a pumping speed at the tube port of at least 25 l. per sec.

(*b*) Adequate precautions, such as a cold trap, should be employed to avoid presence of slight amounts of mercury or oil vapors in the manifold.

(*c*) After the standard diodes are placed on the manifold and reach a vacuum of at least 50 μ of mercury, they shall be baked out by a surrounding oven for at least 15 min. at 400 C. or a temperature just below the softening point of the glass.

(*d*) Degassing and cathode breakdown schedule:

(*1*) Bomb plate for 3 min. (800 C. brightness temperature at the plate center),

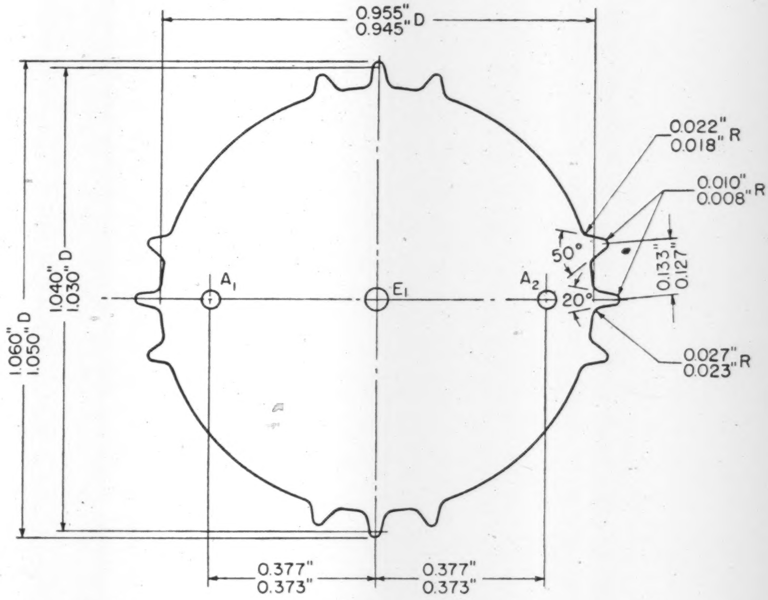
(*2*) Breakdown cathode carbonate coating for 1 min. at 15 v. E_f applied gradually,

(*3*) Bomb plate for 2 min., while applying 9.0 v. E_f,

(*4*) Flash getter, while applying 9.0 v. E_f, and

(*5*) Tip off.

NOTE.—For a Batalum getter apply 1-min. degassing (dull red) for a minimum of ½ min., preceding step (*1*).



NOTE.—Centers of all holes on same center line must not deviate more than 0.001 in. from that line.
A holes—0.041 in. to 0.040 in. diameter.
E hole—0.047 in. to 0.046 in. diameter.
Mica—0.008 in. to 0.012 in. thick.

FIG. 10.—Mica Spacer.

Seasoning (Automatic)

A17. A typical seasoning schedule to accompany automatic exhaust is as follows:

Step	Time, min.	E_f, v.	E_B v., d.c.
1......	1	10.0	...
2......	10	12.5	100
3......	10	10.0	100
4......	10	10.0	...

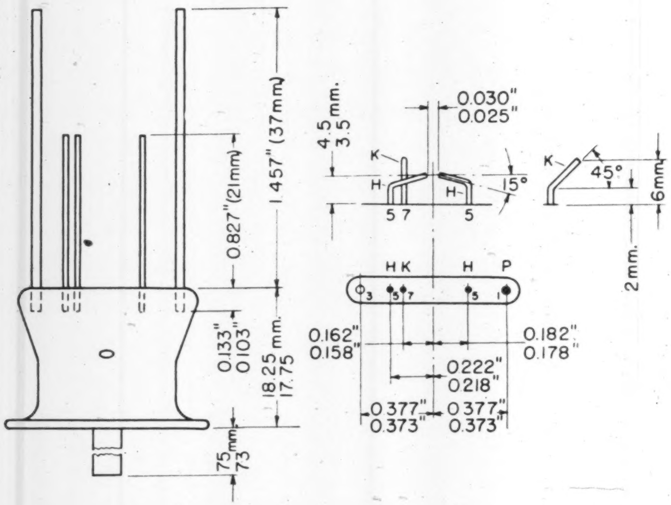
NOTE.—Tungsten filament lamps of 10 w., 120 v. shall be used in the plate circuit in series with each tube during steps 2 and 3. Cold d-c. resistance of the lamps should be matched within ± 5 ohms.

Seasoning (Manual)

A18. The following schedule is recommended for use in connection with the stationary exhaust schedule of Section A15:

Step	E_f, v.	E_B, v., d.c.	Time, min.	Average I_p, ma.
Preheat.....	6.5	...	6	..
1.....	10.0	...	1	..
2.....	12.5	100	12	38
3.....	10.0	100	9	35
4.....	10.0	...	10	..

NOTE.—Tungsten filament lamps of 6 w., 120 v. shall be used in the plate circuit in series with each tube during steps 2 and 3. Cold d-c. resistances of the lamps should be matched within ± 5 ohms (in about 205 ohms). E_p (effective) is approximately 28 v.


0.827" (21mm)
1.457" (37mm)
0.133"
0.103"
18.25 mm.
17.75
75 mm
73
4.5 mm.
3.5
0.030"
0.025"
K
H
H
15°
5 7
5
K
45°
6mm
2mm.
H K
H P
3
5
7
5
1
0.162"
0.158"
0.182"
0.178"
0.222"
0.218"
0.377"
0.373"
0.377"
0.373"

1 Plate lead 4040 Ni-1612D-2039 Cu.
3 Plate dummy 4040 Ni-163D
5 Heater lead 2024 Ni-168D-2054 Cu.
7 Cathode lead 2024 Ni-168D-2044 Cu.

FIG. 11.—Stem Assembly.

SUPPLEMENT II

INSTRUCTIONS FOR USING REPORT FORMS

The report forms shall be used as follows:

A1. *Data for Tubes Tested* (*Table I*):

1. Static Emission at $E_f = 6.5$ v. a.c. or d.c., $E_s = 40$ v. d.c.
2. Static Emission at $E_f = 4.5$ v. a.c. or d.c., $E_s = 40$ v. d.c.
3. Peak Emission ($\hat{I}_s$) at $E_f = 4.5$ v. a.c. or d.c., $E_s = 1000$ v. Pulse width $= 1 \mu$ sec. Repetition rate = 60 to 200 pps. (as available). Report pulse conditions actually used at bottom of page.
4. Low field emission ($\underset{\vee}{I_s}$) at $E_f = 1.75$ v. d.c., $E_s = 4.0$ v. d.c.
5. Filament current at $E_f = 6.5$ v. d.c. (preferred) or a.c. or Brightness Temperature at $E_f = 7.5$ v. a.c. or d.c. Use optical pyrometer of hot disappearing wire type focused upon bare cathode between top of plate and top mica; or focused through sighting hole in center of plate and upon cathode coating. Report in notes at bottom of page location of temperature reading.
6. (*a*) Figure of merit for d-c emission (see method of Section 14). (*b*) Quality ratio for d-c. emission.

Note—$\bar{X}$ = Average; R = Range; r = Ratio of $\bar{X}$ test lot to $\bar{X}$ control lot.

A2. *Cathode Data* (*Table I*):

The following symbols shall be used:

Coating Texture: S = Smooth; M = Medium; R = Rough.

Coating Hardness: S = Soft; M = Medium; H = Hard.

Interface: 0 = None; VL = Very Light; L = Light; A = Average; VH = Very Heavy; H = Heavy.

Plate Barrel Color: I = Iridescent; B = Blue; Br = Brown; Bl = Black; G = Grey. (Prefix Body Color with B, Ring Color with R)

Dome Deposit: 0 = None; L = Light; M = Medium; H = Heavy or report average diameter of deposit in mm.

A3. *Plotting* (*Table I*).—Construct and append copy of $E_f - I_s$ curve for each test and control for $E_s = 40$ v. and E_f between 3.0 v. and 7.0 v. using 1 cycle log-log paper. Construct tangent line per Section 14.

A4. *Processing*.—Cathode, plate, mica and heater processing shall be given in the form for reporting general data (Table II).

TABLE I.—ILLUSTRATIVE FORM FOR REPORTING INITIAL TEST DATA.

Company:.. **Date:**..............................

I. Test Identification:

Lot No.	Alloy	Melt	Other Special Features
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....
....	....	....	....

Lot No.	....	....	....	....	....	....
II. (*a*) Exhaust date.	....	....	....	....	....	....
(*b*) Number tubes tested:						
1. I_s 6.5 $\bar{X}$	....	....	....	....	....	....
R	....	....	....	....	....	....
r	....	....	....	....	....	....
2. I_s 4.5 $\bar{X}$	....	....	....	....	....	....
R	....	....	....	....	....	....
r	....	....	....	....	....	....
3. $\hat{I}_s$ 4.5 $\bar{X}$	....	....	....	....	....	....
R	....	....	....	....	....	....
r	....	....	....	....	....	....
4. $\underset{\vee}{I_s}$ 1.75 $\bar{X}$	....	....	....	....	....	....
R	....	....	....	....	....	....
r	....	....	....	....	....	....
5. I_f 6.5 $\bar{X}$	....	....	....	....	....	....
or KT deg. Cent. R.	....	....	....	....	....	....
6. DC. Emission:						
Figure of Merit	....	....	....	....	....	....
Quality Ratio	....	....	....	....	....	....

(*c*) Pulse conditions: Width........ **μSec**........ **Repetition rate**...............

Pulse shape..

(*d*) Cathode temperature read at $\begin{pmatrix}\text{top}\\ \text{center}\end{pmatrix}$ of plate..

TABLE I.—(*Continued*)

III. Cathode:						
(*a*) Coating mix						
(*b*) Coating date						
(*c*) Coating weight, mg.						
(*d*) Coating diameter, mils.						
(*e*) Coating weight per unit area, mg. per sq. cm.						
(*f*) Coating density, mg. per cu. mm.						
IV. Analysis Before Life (at least 2 tubes per lot):						
(*a*) Plate diameter, mils.						
(*b*) Coated K (cathode) diameter, mils						
(*c*) P-K (plate-cathode coating) spacing, mils.						
(*d*) Plate ID (inside diameter) color						
(*e*) Cathode coating texture						
(*f*) Cathode coating hardness						
(*g*) Cathode interface color						
(*h*) Dome deposit						
V. Analysis after Life (at least 2 tubes per lot):						
(*a*) Plate diameter, mils.						
(*b*) Coated K (cathode) diameter, mils						
(*c*) P-K (plate-cathode coating) spacing, mils						
(*d*) Plate ID (inside diameter) color						
(*e*) Cathode coating texture						
(*f*) Cathode coating hardness						
(*g*) Cathode interface color						
(*h*) Dome deposit						

TABLE II.—ILLUSTRATIVE FORM FOR REPORTING GENERAL DATA.

Company:.. Date:..............................

These data apply to test lots numbered:.............. ....

I. Cathode Processing:

(*a*) Cleaning procedure..

(*b*) Firing temperature........... Time........... Atmosphere..

II. Plate Processing:

(*a*) Cleaning procedure..

(*b*) Firing temperature........... Time........... Atmosphere..

III. Mica Processing:

(*a*) Cleaning procedure..

(*b*) Firing temperature........... Time........... Atmosphere..

IV. Heater Processing:

(*a*) Type of heater used..

(*b*) Temperature of highest firing..

V. Getter:

Type.................. Size.................. Make.................. Lot No..................

VI. Bulb and Stem Cleaning Methods:

....

VII. Exhaust:

(*a*) Type of machine.................. Index speed..................

(*b*) Schedule..

VIII. Seasoning:

Step	E_f	E_b	E_{hk}	Time
1				
2				
3				
4				
5				

Supply volts.......... Plate lamp.......... HK lamp..........

IX. Remarks:

....

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT[1]

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year: at New York, N. Y., on October 6, 1949, and at Philadelphia, Pa., on January 25, 1950. At these sessions the Advisory Committee and Subcommittees W-1, W-2, W-3, W-4, G-1 and G-3 also met.

During the year 7 members were added to the committee and there were 12 removals. At the present time the committee consists of 133 members, of whom 113 are voting members; 51 are classified as producers, 45 as consumers, and 17 as general interest members.

The committee records with sorrow the death of Carter S. Cole on November 17, 1949. Mr. Cole had been a member of the committee for many years; he had been first vice-chairman from 1944 to 1946 and secretary of Subcommittee F-1 since 1944. His personal qualities, his diligence, his expert knowledge of copper alloys and his position as a member of the Headquarters staff of the Society provided assistance which is sorely missed.

At the meeting on January 25, 1950, the election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, G. H. Harnden.
First Vice-Chairman, H. C. Ashley.
Second Vice-Chairman, J. J. Kanter.
Secretary, V. P. Weaver.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is submitting revisions in 14 tentative specifications and revisions in 7 standards for immediate adoption. The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

The following is the report of the several subcommittees during the year. The actions on standards are given in detail in the Appendix.

Subcommittee W-1 on Plate, Sheet, and Strip (S. A. Rosecrans, chairman, W. D. France, secretary) recommended that revisions be incorporated in the following 9 tentative specifications:

A.S.T.M. Designation	Material
B 19	Cartridge Brass
B 36	Brass
B 121	Leaded Brass
B 122	Copper - Nickel - Zinc and Copper-Nickel Alloy
B 130	Gilding Metal
B 152	Copper
B 169	Aluminum Bronze
B 194	Beryllium Copper
B 195	Beryllium Copper, Special Grade

Revisions for immediate adoption were recommended in two standard specifications:

B 97 for Copper-Silicon Alloy Plate, Sheet and Strip for General Purposes and

B 103 for Phosphor Bronze Plate, Sheet and Strip.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The letter ballot vote on these recommendations was favorable: the results of the vote are on record at A.S.T.M. Headquarters.

A task group (W. D. France, chairman) prepared the revisions of chemical compositions in Specifications B 121. Another task group (G. R. Gohn, chairman) prepared the revisions of physical properties in Specifications B 194 and B 195.

Among the subjects being studied by the subcommittee are the addition of sawed bar to plate specifications, the addition of alloy No. B 2 of Specifications B 139 for Phosphor Bronze Rods, Bars, and Shapes to Specifications B 103, the use of Rockwell hardness information as a mandatory requirement in 8 plate, sheet, and strip specifications, the addition of a new alloy to Specifications B 169 and the addition of two new alloys to Specifications B 171 for Copper-Alloy Condenser Tube Plates.

Subcommittee W-2 on Rods, Bars, and Shapes (J. D. MacQueen, chairman) recommended that revisions be incorporated in three tentative specifications: B 21 for Naval Brass, B 139 for Phosphor Bronze, and B 151 for Copper-Nickel-Zinc Alloy. Revisions for immediate adoption were recommended in Standard Specifications B 140 for Leaded Red Brass (Hardware Bronze).

Among the subjects being considered in the subcommittee are the inclusion of additional size ranges in Specifications B 133 for Copper Rods, Bars, and Shapes, the collection of Rockwell hardness data in anticipation of future inclusion of such requirements in rod and bar specifications, the addition of a new alloy to Specifications B 150 for Aluminum Bronze, the revision of requirements for mechanical properties in Specifications B 196 for Beryllium Copper, and the possibility of specifications for forgings.

Subcommittee W-3 on Wire and Wire Rod (B. J. Sirois, chairman) recommended that revisions be incorporated in two tentative specifications: B 197 for Beryllium Copper and B 206 for Copper-Nickel-Zinc Alloy. Revisions for immediate adoption were recommended in Standard Specifications B 134 for Brass.

A task group (H. S. Freynik, chairman) prepared the revisions for tensile requirements in Specifications B 197. Another task group (W. D. France, chairman) prepared the revision of lot size for sampling for chemical analysis in Specifications B 134.

Subcommittee W-4 on Pipe and Tube (Alan Morris, chairman, R. S. Pratt, secretary) recommended revisions for immediate adoption in two standard specifications: B 88 for Seamless Copper Water Tube and B 111 for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock.

Among the subjects under consideration in the subcommittee are the development of requirements for automotive and refrigeration service tube, review of temper requirements in Specifications B 75 for Seamless Copper Tubes, review of Rockwell hardness requirements in tube specifications and the addition of 90-10 copper-zinc alloy to Specifications B 135 for Seamless Brass Tubes. For Specifications B 111, the subcommittee is studying the revision of dimensional tolerances, the addition of requirements for drawn temper for copper-nickel alloys, and the addition of a copper-nickel-aluminum alloy.

Subcommittee F-1 on Castings, and Ingots for Remelting (G. H. Clamer, chairman) is reactivating the task group to review Specifications B 61 for Steam or Valve Bronze and B 62 for Composition Brass or Ounce Metal and is studying the preparation of a document similar to the "Code of Procedure in Inspection of Copper-Base Alloy Sand Castings" adopted by the British Standards Institution in 1947. In cooperation with Committee E-3 the subcommittee is studying the revision of provisions for sampling for chemical analysis in specifications for ingots and castings.

Subcommittee G-1 on Methods of Test (G. R. Gohn, chairman, J. P. Guerard, secretary) recommended revisions for immediate adoption in the Standard Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 - 45).

The subcommittee is continuing its studies on the effect of speed of testing on tensile properties, the relation of Rockwell hardness to thickness of specimen, and the revision of Standard Method B 154.

Committee E-1 on Methods of Testing has under consideration a revision of the Standard Methods of Tension Testing of Metallic Materials (E 8 - 46) to incorporate the requirements now included in the Tentative Methods of Tension Testing of Copper and Copper-Alloy Rods, Bars, and Shapes (B 220 - 48 T).

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman) recommended the revisions of straightness tolerances for slit materials in 11 plate, sheet, and strip specifications.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) is continuing the study of nomenclature and classification of wrought alloys under the jurisdiction of the committee.

This report has been submitted to letter ballot of the committee, which consists of 113 voting members; 92 members returned their ballots, of whom 89 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. H. Harnden,
Chairman.

V. P. Weaver,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1949 Book of A.S.T.M. Standards, Part 2.

Revisions of Tentative Specifications

The committee recommends that the following 14 tentative specifications be revised as indicated below and continued as tentative.

Tentative Specifications for Cartridge Brass Sheet, Strip, Plate, Bar and Disks (B 19 - 49 T):

Table X.—Revise the portion headed "Slit Metal" to read as follows:

Width, in.	Straightness Tolerance, in.		
	As Slit Only		As Slit and Either Straightened or Edge Rolled
	Shipped in Rolls	Shipped Flat	Shipped Flat, in Rolls, or on Bucks
Over ¼ to ⅜, incl.	3	1½	½
Over ⅜ to ½, incl.	2	1	½
Over ½ to 1, incl.	1¼	¾	½
Over 1 to 2, incl.	¾	⅝	½
Over 2 to 4, incl.	⅝	½	½
Over 4	½	½	½

Tentative Specifications for Naval Brass Rods, Bars, and Shapes (B 21 - 49 T):

Table I.—For alloy C change the lead content range from the present "1.25 to 2.25" to read "1.3 to 2.2" per cent.

Tentative Specifications for Brass Plate, Sheet, and Strip (B 36 - 49 T):

Table VIII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Specifications B 19.

Tentative Specifications for Leaded Brass Plate, Sheet, and Strip (B 121 - 49 T):

Section 1 (a).—Revise the table of nominal compositions to read as follows, indicating that six instead of five alloys are specified, and delete Footnote *a*:

Alloy No.	Nominal Composition		
	Copper, per cent	Lead, per cent	Zinc, per cent
1	90.0	0.5	9.5
2	65.0	0.5	34.5
3	65.0	1.0	34.0
4	63.0	1.8	35.2
5	65.0	2.0	33.0
6	63.0	2.5	34.5

Section 4.—Delete Footnote *a*, and revise the table of chemical compositions to read as follows:

Alloy No.	Copper, per cent	Lead, per cent	Iron, max., per cent	Zinc, per cent
1	89.0 to 91.0	0.3 to 0.7	0.10	remainder
2	62.5 to 66.5	0.3 to 0.7	0.10	remainder
3	62.5 to 66.5	0.8 to 1.4	0.10	remainder
4	60.5 to 64.5	1.3 to 2.3	0.10	remainder
5	62.5 to 66.5	1.5 to 2.5	0.10	remainder
6	60.5 to 64.5	2.0 to 3.0	0.10	remainder

Tables I, II, and III.—Add alloy No. 5 to the group designated alloy Nos. 2, 3, 4 and 6.

Table VIII.—Revise the portion headed "Slit Metal and Slit Metal with

Rolled Edges" as indicated above for Table X in Specifications B 19.

Tentative Specifications for Copper-Nickel-Zinc and Copper-Nickel Alloy Plate, Sheet, and Strip (B 122 - 49 T):

Table VIII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Specifications B 19.

Tentative Specifications for Gilding Metal Strip (B 130 - 49 T):

Table VIII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Specifications B 19.

Tentative Specifications for Phosphor Bronze Rods, Bars, and Shapes (B 139 - 49 T):

Table I.—In the last line of the first column and in Footnote *a* delete the words "plus lead".

Add a new Footnote *b* referenced after the value "99.5" for alloy B 1 to read "[b] For alloy B 1 the minimum percentage of 99.5 includes lead content as well as copper plus tin plus phosphorus."

Reletter the present Footnote *b* as *c*, adding the words "lead and" after the word "includes" and delete the words "plus lead."

Tentative Specifications for Copper-Nickel-Zinc Alloy Rods and Bars (B 151 - 49 T):

Table I.—For alloy C change the lead content range from the present "0.75 to 1.25" to read "0.8 to 1.2" per cent.

Tentative Specifications for Copper Sheet, Strip, and Plate (B 152 - 49 T):

Table IX.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Specifications B 19.

Tentative Specifications for Aluminum Bronze Plate, Sheet, and Strip (B 169 - 49 T):

Table VII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Specifications B 19.

Tentative Specifications for Beryllium-Copper Alloy Plate, Sheet, and Strip (B 194 - 49 T):

Table II.—Revise to read as shown in the accompanying Table I.

TABLE I.—MECHANICAL TEST REQUIREMENTS FOR COLD-ROLLED PLATE, SHEET AND STRIP AS SUPPLIED COMMERCIALLY.

Condition	Tensile Strength, psi.		Rockwell Hardness[a]			
			B scale		Superficial 30 T Scale	
	min.	max.	min.	max.	min.	max.
A.....	65 000	78 000	...	79	...	67.0
¼ H....	76 000	88 000	74	90	64.0	75.5
½ H....	86 000	102 000	89	96	74.5	79.5
H.....	105 000	125 000	97	102	80.5	83.5

[a] The minimum thickness of material that may be tested in the case of the above Rockwell hardness scales is as follows:

	Thickness, in.
B Scale	0.032 and over
Superficial 30 T Scale	0.010 and over

Table III.—Delete all of the present Rockwell hardness requirements and replace with the following, revising the present Footnote *b* as indicated:

Condition	Rockwell Hardness[b], min C Scale	Rockwell Hardness[b], min Superficial 30 N Scale
AT	30	52.5
¼ HT	33	55.0
½ HT	36	57.5
HT	38	59.5

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[b] The minimum thickness of material that may be tested in the case of the above Rockwell hardness scales is as follows:

	Thickness, in.
C Scale	0.032 and over
Superficial 30 N Scale	0.010 and over

Table VIII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Specifications B 19.

New Appendix.—Add an Appendix II to read "Table IX (accompanying Table II) shows the approximate relationship between tensile strength and Vickers diamond pyramid hardness. The tensile values given in the table are those specified as limiting values in Tables II and III."

Tentative Specifications for Beryllium-Copper Alloy Strip, Special Grade (B 195 - 49 T):

Table II.—Revise to read as shown in the accompanying Table I except the title shall read "Mechanical Test Requirements for Cold-Rolled Strip (Special Grade) As Supplied Commercially."

TABLE II.—APPROXIMATE RELATIONSHIP BETWEEN TENSILE STRENGTH AND VICKERS DIAMOND PYRAMID HARDNESS.

Tensile Strength, psi.	Vickers Diamond Pyramid Hardness[a]	
	2½-Kg. Load	5-Kg. Load
65 000	...	...
76 000	149	142
78 000	163	157
86 000	200	195
88 000	207	201
102 000	240	233
105 000	245	238
125 000	274	271
150 000	334	335
160 000	355	356
170 000	371	373
180 000	385	387
185 000	391	394

[a] Applies to thicknesses of 0.010 in. and over.

Table III.—Add the following hardness requirements:

Condition	Rockwell Hardness[b], min. C Scale	Rockwell Hardness[b], min. Superficial 30 N Scale
AT	33.5	55.0
¼ HT	35.5	57.5
½ HT	37.5	59.5
HT	38.5	60.0

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[b] The minimum thickness of material that may be tested in the case of the above Rockwell hardness scales is as follows:

	Thickness, in.
C Scale	0.032 and over
Superficial 30 N Scale	0.010 and over

Table VIII.—Revise as indicated above for Table X in Specifications B 19, omitting the last line of Table X.

New Appendix.—Add an Appendix to read as indicated above for new Appendix II in Specifications B 194.

Tentative Specifications for Beryllium-Copper Alloy Wire (B 197 - 49 T):

Table III.—Revise to read as shown in the accompanying Table III.

TABLE III.—MECHANICAL TEST REQUIREMENTS FOR WIRE AFTER PRECIPITATION HARDENING.

Condition	Tensile Strength, psi.
AT	160 000 to 190 000
¼ HT	175 000 to 205 000
½ HT	185 000 to 215 000
¾ HT[a]	190 000 to 220 000

[a] Three-quarter hard temper is generally available in diameters up to 0.080 in., inclusive.

Tentative Specifications for Copper-Nickel-Zinc Alloy Wire (B 206 - 49 T):

Table I.—For alloy C change the lead content range from the present "0.75 to 1.25" to read "0.8 to 1.2" per cent.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends, for immediate adoption, revisions in 7 specifications as indicated below and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these modifications may be referred to letter ballot of the Society.

Standard Specifications for Seamless Copper Water Tube (B 88 - 49):

Section 1.—In the fourth line after the word "services" add the word "drainage."

Section 3.—For type M add "Sizes 1¼, 1½, and 2 in. are suitable solely for soil, waste, and vent lines, and other interior non-pressure uses."

Table II.—Revise by the addition of the sizes shown in the accompanying Table IV.

Under the headings for types K, L, and M add the following:

"Type K.—Recommended for underground service, general plumbing and heating purposes involving severe service conditions, and for gas, steam and oil lines.

Type L.—Recommended for interior applications involving general plumbing and heating purposes.

Type M.—
- Sizes 2½ to 12 in., inclusive, recommended for use with soldered fittings only, for interior applications involving general plumbing and heating purposes.
- Sizes 1¼ to 12 in., inclusive, recommended for use with soldered fittings only, for waste, vent, and soil lines, and for other interior non-pressure applications."

TABLE IV.—ADDITIONAL SIZES OF SEAMLESS COPPER WATER TUBE.

Standard Water Tube Size, in.	Actual Outside Diameter, in.	Average Outside Diameter Tolerance, in.		Wall Thickness, in.				Theoretical Weight, lb. per ft.	
				Type L		Type M		Type L	Type M
		Annealed	Drawn	Nominal	Tolerance	Nominal	Tolerance		
¼	0.375	0.002	0.001	0.030	0.0035	...	...	0.126	...
1¼	...	...	...	...	...	0.042	0.0035	...	0.682
1½	...	...	...	...	...	0.049	0.004	...	0.940
2	...	...	...	...	...	0.058	0.006	...	1.46

Section 15(a).—Revise "20 ft." to read "either 12 ft. or 20 ft."

Standard Specifications for Copper-Silicon Alloy Plate, Sheet and Strip for General Purposes (B 97 - 49):

Table VIII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Tentative Specifications B 19.

Standard Specifications for Phosphor Bronze Plate, Sheet, and Strip (B 103 - 49):

Table I.—In the sixth line of the first column delete the words "plus lead."

Add a Footnote *a* referenced after the value "99.5" for alloys B and B-1 to read "[a] For alloys B and B-1 the minimum percentage of 99.5 includes lead content as well as copper plus tin plus phosphorus."

Table VII.—Revise the portion headed "Slit Metal and Slit Metal with Rolled Edges" as indicated above for Table X in Tentative Specifications B 19.

Standard Specifications for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 - 49):

Table I.—For aluminum brass delete Footnote *c* and reletter subsequent footnotes accordingly.

For copper-nickel alloys add minimum copper content as follows; also retain footnote *f*:

70–30 copper-nickel	65.0
80–20 copper-nickel:	
Type A	74.0
Type B	70.0
90–10 copper-nickel	86.5

For 90-10 copper-nickel alloy change the iron content from the present "1.0 max." to read "0.5 to 2.0" per cent.

Standard Specifications for Brass Wire (B 134 - 49):

Section 5.—Change the present lot size "10,000 lb." to read "5000 lb."

Standard Specifications for Leaded Red Brass (Hardware Bronze) Rods, Bars, and Shapes (B 140 - 49):

Section 4.—For alloy A change the lead content range from the present "1.50 to 2.25" to read "1.5 to 2.2" per cent. For alloy B change the lead content range from the present "1.25 to 2.25" to read "1.3 to 2.2" per cent.

Standard Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 - 45):

Section 1.—Revise the second sentence to read "This test is an accelerated corrosion test for the purpose of determining in copper or copper-base alloy products, the presence of residual (internal) stresses which might bring about failure of the material in service or storage through season cracking."

Revise the beginning of Note 1 to read "The following definitions are supplied for information."

Add a new Note 2 to read "This method was originally prepared to cover the mercurous nitrate testing of certain mill products supplied in accordance with A.S.T.M. Committee B-5 specifications. While the method of test has also been used for testing assemblies and partial assemblies, the method was not written with that intent and some modification may be required for such use."

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS[1]

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: one in Atlantic City, N. J., on June 29, 1949, and one in Pittsburgh, Pa., on March 1, 1950, with an attendance of 31 and 45, respectively.

Subcommittee IV on Physical Tests on Die Castings has been discharged since it has completed its agenda.

There is a balance of $2642.29 in the funds of Committee B-6, as of December 31, 1949.

At the present time, the committee consists of 92 members, of whom 43 are classified as producers, 23 as consumers, and 26 as general interest members.

REVISION OF TENTATIVE

The committee recommends a revision in the following tentative specification:

Tentative Specifications for Aluminum-Base Alloy Die Castings (**B 85 - 49 T**):[2]

Table I.—Change the maximum limit for zinc in alloys SC5 and SC6 from 0.6 per cent to 1.0 per cent.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the *Tentative Specifications for Copper-Base (Brass) Alloy Die Castings* (**B 176 - 49 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (J. J. Bowman, chairman) recommended, in view of the favorable results obtained by a Task Group on the study of the effect of zinc contamination on the properties of SC5 and SC6, that the maximum zinc content for these alloys as given in Specifications B 85 - 49 T be raised from 0.6 to 1.0 per cent.

Subcommittee V on Exposure and Corrosion Tests (W. M. Peirce, chairman) completed the testing of the magnesium-base and zinc-base alloys from five outdoor and three indoor sites after 10 yr. of exposure. The results of the original properties of these alloys and the 5-yr. aging data were included in the 1939[4] and 1946 reports,[5] respectively. The 10-yr. data are given in Table I appended to this report.

The remaining lot of magnesium-base and zinc-base alloy specimens will be left at these eight sites until 1959 for a total exposure of 20 yr.

Subcommittee IX on Die-Casting Process (A. E. Martin, chairman) reported

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 2, p. 579.

[3] The letter ballot vote on these recommendations was favorable: the results of the vote are on record at A.S.T.M. Headquarters.

[4] *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 280 (1939).

[5] Ibid., Vol. 46, p. 244.

TABLE I.—MECHANICAL PROPERTY DATA ON MAGNESIUM-BASE AND ZINC-BASE ALLOY SPECIMENS AFTER 10-YR. ATMOSPHERIC EXPOSURE.

Alloy No.	Producer	Analyzed Composition, per cent	Exposure		Tensile Strength, psi.[c]	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Rockwell "E" Hardness	Dimensional Change, in. per 6 in.
			Type	Location					
XXIa	A	Al....4.2 Cu...2.8 Mg...0.04	Outdoors	Key West, Fla.	[a]	1	[a]	95	0.0110
				Sandy Hook, N. J.	47 800	6	3	100[b]	0.0031
				New York, N. Y.	44 000	9	3	97	0.0042
				Altoona, Pa.	41 900	9	1	81	0.0048
				State College, Pa.	46 500	6	3	92	0.0024
			Indoors	Coco Solo, C. Z.	46 200	3	4	95	0.0102
				New Kensington, Pa.	50 900	4	4	94	0.0058
				Tucson, Ariz.	48 300	3	3	97	0.0093
XXIa	D	Al....3.8 Cu...2.8 Mg...0.05	Outdoors	Key West, Fla.	44 800	1	2	96	0.0117
				Sandy Hook, N. J.	47 300	2	2	106[b]	0.0046
				New York, N. Y.	44 000	3	1	100	0.0052
				Altoona, Pa.	42 300	5	1	84	0.0057
				State College, Pa.	47 800	2	1	93	0.0034
			Indoors	Coco Solo, C. Z.	44 900	3	3	93	0.0107
				New Kensington, Pa.	49 100	4	3	90	0.0066
				Tucson, Ariz.	48 500	4	2	98	0.0085
XXIa	J	Al....4.2 Cu...2.8 Mg...0.05	Outdoors	Key West, Fla.	[a]	1	[a]	96	0.0076
				Sandy Hook, N. J.	48 700	4	3	106[b]	0.0014
				New York, N. Y.	43 200	6	3	98	0.0042
				Altoona, Pa.	41 700	8	1	80	0.0017
				State College, Pa.	47 200	6	3	83	0.0005
			Indoors	Coco Solo, C. Z.	47 200	3	3	93	0.0070
				New Kensington, Pa.	52 000	5	5	90	0.0026
				Tucson, Ariz.	49 500	4	2	97	0.0053
XXIa	P	Al....3.8 Cu...2.6 Mg...0.05	Outdoors	Key West, Fla.	[a]	4	[a]	95	0.0081
				Sandy Hook, N. J.	44 900	13	5	100[b]	0.0008
				New York, N. Y.	39 800	15	4	97	0.0027
				Altoona, Pa.	39 100	18	2	82	0.0032
				State College, Pa.	42 900	11	4	91	0.0004
			Indoors	Coco Solo, C. Z.	42 700	4	3	94	0.0064
				New Kensington, Pa.	46 700	20	6	91	0.0024
				Tucson, Ariz.	47 000	7	3	97	0.0051
XXIa	S	Al....4.1 Cu...3.0 Mg...0.06	Outdoors	Key West, Fla.	[a]	1	[a]	96	0.0137
				Sandy Hook, N. J.	54 600	1	2	107[b]	0.0078
				New York, N. Y.	45 200	1	3	101	0.0087
				Altoona, Pa.	48 700	2	1	88	0.0075
				State College, Pa.	53 600	2	2	94	0.0056
			Indoors	Coco Solo, C. Z.	49 000	3	4	96	0.0142
				New Kensington, Pa.	54 100	3	4	94	0.0091
				Tucson, Ariz.	52 600	2	5	97	0.0125
XXIa	Z	Al....4.3 Cu...2.8 Mg...0.04	Outdoors	Key West, Fla.	[a]	1	[a]	96	0.0109
				Sandy Hook, N. J.	51 200	5	5	103[b]	0.0037
				New York, N. Y.	46 100	5	3	101	0.0058
				Altoona, Pa.	45 400	6	2	84	0.0046
				State College, Pa.	50 000	7	4	95	0.0028
			Indoors	Coco Solo, C. Z.	49 800	3	4	96	0.0112
				New Kensington, Pa.	54 600	12	6	94	0.0060
				Tucson, Ariz.	51 700	6	3	98	0.0101
XXIII	A	Al....4.0 Mg...0.05	Outdoors	Key West, Fla.	31 000	[d]	16	76	0.0010
				Sandy Hook, N. J.	33 400	19	14	61[b]	−0.0001
				New York, N. Y.	31 500	32	9	81	−0.0007
				Altoona, Pa.	32 000	43	6	68	−0.0008
				State College, Pa.	33 600	28	9	75	−0.0001
			Indoors	Coco Solo, C. Z.	32 500	42	22	77	−0.0001
				New Kensington, Pa.	34 700	39	20	70	−0.0003
				Tucson, Ariz.	34 200	38	20	79	−0.0005
XXIII	J	Al....4.2 Mg...0.05	Outdoors	Key West, Fla.	[a]	[d]	[a]	75	0.0011
				Sandy Hook, N. J.	33 300	8	10	61[b]	−0.0005
				New York, N. Y.	30 800	31	11	81	−0.0010
				Altoona, Pa.	32 100	44	6	71	−0.0009
				State College, Pa.	33 400	33	7	75	−0.0003

TABLE I.—*Continued*

Alloy No.	Producer	Analyzed Composition, per cent	Exposure		Tensile Strength, psi.[c]	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Rockwell "E" Hardness	Dimensional Change, in. per 6 in.
			Type	Location					
			Indoors	Coco Solo, C. Z.	32 500	43	22	75	−0.0003
				New Kensington, Pa.	34 700	39	13	74	−0.0004
				Tucson, Ariz.	33 800	40	14	79	−0.0006
XXIII	P	Al....3.8 Mg...0.04	Outdoors	Key West, Fla.	31 400	[d]	22	76	0.0021
				Sandy Hook, N. J.	32 800	18	19	61[b]	0.0000
				New York, N. Y.	30 400	33	24	80	−0.0010
				Altoona, Pa.	32 200	43	11	70	−0.0011
				State College, Pa.	33 400	33	15	74	−0.0002
			Indoors	Coco Solo, C. Z.	31 900	44	28	76	−0.0001
				New Kensington, Pa.	34 400	41	25	75	−0.0005
				Tucson, Ariz.	33 700	40	23	79	−0.0007
XXIII	Z	Al....4.1 Mg...0.05	Outdoors	Key West, Fla.	27 500	[d]	16	78	0.0008
				Sandy Hook, N. J.	34 000	24	13	62[b]	−0.0001
				New York, N. Y.	31 600	30	18	82	−0.0006
				Altoona, Pa.	33 700	46	12	73	−0.0006
				State College, Pa.	33 900	21	13	75	−0.0002
			Indoors	Coco Solo, C. Z.	33 100	44	20	78	−0.0001
				New Kensington, Pa.	35 400	40	17	75	−0.0003
				Tucson, Ariz.	34 800	40	17	80	−0.0005
XXV	A	Al....4.1 Cu...1.1 Mg...0.04	Outdoors	Key West, Fla.	36 100		13	85	0.0000
				Sandy Hook, N. J.	39 100	29	8	79[b]	−0.0005
				New York, N. Y.	36 000	27	5	89	0.0003
				Altoona, Pa.	34 000	26	3	75	0.0003
				State College, Pa.	39 000	26	5	83	−0.0008
			Indoors	Coco Solo, C. Z.	36 600	35	17	84	−0.0006
				New Kensington, Pa.	40 000	37	15	83	−0.0012
				Tucson, Ariz.	38 100	31	13	88	−0.0007
XXV	D	Al....3.9 Cu...1.0 Mg...0.05	Outdoors	Key West, Fla.	[a]	[d]	[a]	87	0.0010
				Sandy Hook, N. J.	39 100	30	5	82[b]	−0.0004
				New York, N. Y.	36 800	26	3	90	0.0015
				Altoona, Pa.	35 500	23	2	77	0.0003
				State College, Pa.	39 800	16	3	86	−0.0005
			Indoors	Coco Solo, C. Z.	37 400	35	10	84	−0.0002
				New Kensington, Pa.	40 300	38	11	85	−0.0006
				Tucson, Ariz.	39 600	38	14	88	−0.0006
XXV	J	Al....4.2 Cu...1.1 Mg...0.05	Outdoors	Key West, Fla.	[a]	[d]	[a]	84	0.0003
				Sandy Hook, N. J.	37 300	24	6	74[b]	−0.0011
				New York, N. Y.	34 100	19	4	87	−0.0002
				Altoona, Pa.	32 600	19	3	72	0.0013
				State College, Pa.	37 400	30	5	81	−0.0013
			Indoors	Coco Solo, C. Z.	35 900	32	9	82	−0.0006
				New Kensington, Pa.	38 500	38	10	81	−0.0014
				Tucson, Ariz.	37 600	34	8	86	−0.0010
XXV	P	Al....3.9 Cu...1.0 Mg...0.05	Outdoors	Key West, Fla.	[a]	[d]	[a]	84	−0.0004
				Sandy Hook, N. J.	37 900	27	9	75[b]	−0.0014
				New York, N. Y.	34 800	28	5	87	−0.0003
				Altoona, Pa.	33 700	24	4	73	0.0003
				State College, Pa.	38 100	30	6	83	−0.0019
			Indoors	Coco Solo, C. Z.	36 300	36	17	84	−0.0016
				New Kensington, Pa.	38 800	40	15	82	−0.0019
				Tucson, Ariz.	38 000	38	12	87	−0.0020
XXV	S	Al....4.2 Cu...1.0 Mg...0.06	Outdoors	Key West, Fla.	[a]	[d]	[a]	86	0.0005
				Sandy Hook, N. J.	40 700	24	9	75[b]	−0.0004
				New York, N. Y.	36 500	22	5	89	0.0013
				Altoona, Pa.	36 200	18	3	74	0.0013
				State College, Pa	40 300	28	6	81	−0.0007
			Indoors	Coco Solo, C. Z.	38 100	28	17	86	−0.0001
				New Kensington, Pa	41 300	34	17	83	−0.0007
				Tucson, Ariz.	39 300	29	8	87	−0.0006

(Continued on pages 170 and 171)

TABLE I.—*Continued*

Alloy No.	Producer	Analyzed Composition, per cent	Exposure		Tensile Strength, psi.^{c}	Charpy Impact Strength, ft-lb (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Rockwell "E" Hardness	Dimensional Change, in. per 6 in.^{e}
			Type	Location					
XXV	Z	Al....4.0	Outdoors	Key West, Fla.	35 600	^{d}	15	86	0.0003
		Cu...1.0		Sandy Hook, N. J.	38 700	31	10	75^{b}	−0.0006
		Mg...0.04		New York, N. Y.	35 500	29	6	89	0.0016
				Altoona, Pa.	34 600	24	4	74	0.0013
				State College, Pa.	38 700	18	8	84	−0.0009
			Indoors	Coco Solo, C. Z.	37 000	36	15	85	−0.0002
				New Kensington, Pa.	39 700	42	20	83	−0.0009
				Tucson, Ariz.	38 700	38	12	87	−0.0007
307	H	Al....6.3	Outdoors	Key West, Fla.	27 700	1	3	..	0.0002
		Si....0.2		Sandy Hook, N. J.	22 700	2	3	65	0.0017
		Mn...0.14		New York, N. Y.	19 600	2^{f}	4	57	0.0124
				Altoona, Pa.	17 200	1^{f}	3	64	0.0244
				State College, Pa.	24 700	2	3	67	0.0018
			Indoors	Coco Solo, C. Z.	28 300	2	5	64	0.0030
				New Kensington, Pa.	30 600	3	5	63	0.0005
				Tucson, Ariz.	29 900	3	4	43^{b}	−0.000
307	K	Al....6.6	Outdoors	Key West, Fla.	30 400	2	5	68	0.000
		Si....0.1		Sandy Hook, N. J.	27 100	2	5	63	0.0011
		Mn...0.24		New York, N. Y.	20 500	2^{f}	5	59	0.0084
				Altoona, Pa.	19 100	1^{f}	4	61	0.0132
				State College, Pa.	28 200	3	6	69	0.0002
			Indoors	Coco Solo, C. Z.	29 300	4	7	64	0.0028
				New Kensington, Pa.	31 100	4	5	64	0.0011
				Tucson, Ariz.	32 900	3	6	38^{b}	0.0001
307	M	Al...6.5	Outdoors	Key West, Fla.	25 300	3	3	..	0.0005
		Si....0		Sandy Hook, N. J.	22 600	3	2	64	0.0006
		Mn ..0.19		New York, N. Y.	17 700	2^{f}	2	56	0.0069
				Altoona, Pa.	15 800	1^{f}	3	59	0.0143
				State College, Pa.	23 400	3	2	67	0.0010
			Indoors	Coco Solo, C. Z.	25 900	3	4	63	0.0026
				New Kensington, Pa.	26 400	4	3	64	0.0010
				Tucson, Ariz.	26 100	5	3	41^{b}	−0.0003
312	H	Al... 8.9	Outdoors	Key West, Fla.	28 400	1	2	..	−0.0003
		Si... 0.2		Sandy Hook, N. J.	25 800	1	2	73	0.0009
		Mn...0.14		New York, N. Y.	20 600	1^{f}	2	76	0.0090
				Altoona, Pa.	19 700	1^{f}	1	71	0.0201
				State College, Pa.	29 200	1	2	79	0.0009
			Indoors	Coco Solo, C. Z.	28 700	1	3	76	0.0021
				New Kensington, Pa.	29 700	1	2	75	0.0002
				Tucson, Ariz.	29 800	1	2	47^{b}	−0.0008
312	K	Al....9.4	Outdoors	Key West, Fla.	30 100	1	2	78	0.0004
		Si....0.3		Sandy Hook, N. J.	26 200	1	2	75	0.0016
		Mn.. 0.24		New York, N. Y.	23 800	1^{f}	3	76	0.0044
				Altoona, Pa.	21 700	0^{f}	3	72	0.0126
				State College, Pa.	30 100	1	2	75	0.0020
			Indoors	Coco Solo, C. Z.	32 800	2	4	76	0.0021
				New Kensington, Pa.	33 300	2	3	73	0.0013
				Tucson, Ariz.	34 100	2	2	45^{b}	0.0001
312	M	Al..10.3	Outdoor	Key West, Fla.	30 700	1	1	..	−0.0002
		Si . 0.4		Sandy Hook, N. J.	27 200	1	1	79	0.0017
		Mn.. 0.14		New York, N. Y.	23 000	1^{f}	1	80	0.0119
				Altoona, Pa.	21 100	1^{f}	1	80	0.0141
				State College, Pa.	31 100	1	1	83	0.0017
			Indoors	Coco Solo, C. Z.	33 400	1	2	83	0.0020
				New Kensington, Pa	31 800	1	1	82	0.0014
				Tucson, Ariz.	32 300	1	2	46^{b}	−0.0005
313	H	Al.. 8.5	Outdoors	Key West, Fla.	28 000	1	2	79	−0.0001
		Si... 0.2		Sandy Hook, N. J.	26 300	1	2	73	0.0017
		Mn...0.14		New York, N. Y	19 800	0^{f}	2	67	0.0102
		Zn.. 0.4		Altoona, Pa.	18 300	1^{f}	2	67	0.0245
				State College, Pa.	28 300	1	2	76	0.0006
			Indoors	Coco Solo, C. Z.	30 300	1	3	76	0.0028
				New Kensington, Pa.	29 000	2	2	73	0.0010
				Tucson, Ariz.	28 800	2	3	36^{b}	−0.0004

TABLE I.—*Continued*

Alloy No.	Producer	Analyzed Composition, per cent	Exposure		Tensile Strength, psi.[c]	Charpy Impact Strength, ft-lb. (¼ by ¼-in. Bars)	Elongation in 2 in., per cent	Rockwell "E" Hardness	Dimensional Change, in. per 6 in.[e]
			Type	Location					
313	K	Al....8.6	Outdoors	Key West, Fla.	31 000	1	3	74	0.0001
		Si....0		Sandy Hook, N. J.	27 600	1	2	74	0.0010
		Mn...0.22		New York, N. Y.	23 400	1[f]	3	73	0.0107
		Zn...0.6		Altoona, Pa.	21 100	1[f]	4	72	0.0249
				State College, Pa.	30 800	1	3	77	0.0021
			Indoors	Coco Solo, C. Z.	33 100	2	6	74	0.0026
				New Kensington, Pa.	32 900	2	4	72	0.0010
				Tucson, Ariz.	32 600	2	3	42[b]	0.0000
313	M	Al....8.6	Outdoors	Key West, Fla.	26 700	1	2	..	0.0002
		Si....0		Sandy Hook, N. J.	26 500	1	3	74	0.0005
		Mn...0.18		New York, N. Y.	22 100	1[f]	3	73	0.0075
		Zn...0.5		Altoona, Pa.	17 300	1[f]	2	72	0.0201
				State College, Pa.	29 200	1	3	75	0.0004
			Indoors	Coco Solo, C. Z.	27 800	2	2	75	0.0030
				New Kensington, Pa.	28 800	2	2	75	0.0008
				Tucson, Ariz.	30 200	2	2	49[b]	−0.0003
314	H	Al... 6.9	Outdoors	Key West, Fla.	31 100	1	3	77	0.0001
		Si....0.2		Sandy Hook, N. J.	23 200	1	2	71	0.0008
		Mn...0.14		New York, N. Y.	17 600	1[f]	2	70	0.0122
		Zn...1.8		Altoona, Pa.	15 800	1[f]	1	67	0.0189
				State College, Pa.	26 100	1	2	74	0.0017
			Indoors	Coco Solo, C. Z.	31 000	2	3	76	0.0025
				New Kensington, Pa.	32 200	2	3	73	0.0010
				Tucson, Ariz.	31 500	2	2	51[b]	−0.0007
314	K	Al....6.4	Outdoors	Key West, Fla.	[a]	2	[a]	..	0.0008
		Si....0		Sandy Hook, N. J.	27 600	2	4	71	0.0015
		Mn...0.33		New York, N. Y.	17 400	1[f]	3	70	0.0062
		Zn...2.3		Altoona, Pa.	17 600	1[f]	4	64	0.0102
				State College, Pa.	30 400	2	6	74	0.0018
			Indoors	Coco Solo, C. Z.	33 500	3	7	75	0.0027
				New Kensington, Pa.	34 300	4	6	64	0.0013
				Tucson, Ariz.	35 000	2	5	48[b]	0.0003
314	M	Al....6.4	Outdoors	Key West, Fla.	24 200	1	2	..	0.0009
		Si....0		Sandy Hook, N. J.	23 500	2	3	70	0.0007
		Mn...0.20		New York, N. Y.	17 700	1[f]	2	66	0.0095
		Zn...2.5		Altoona, Pa.	17 700	1[f]	4	67	0.0141
				State College, Pa.	24 900	1	3	74	0.0017
			Indoors	Coco Solo, C. Z.	31 700	3	3	76	0.0034
				New Kensington, Pa.	31 100	2	3	72	0.0011
				Tucson, Ariz.	30 600	2	2	38[b]	0.0003

[a] Specimens lost.

[b] Brinell hardness.

[c] The original cross-sectional areas were used in computing the tensile strengths.

[d] Specimens did not break in Izod Machine of 60 ft-lb. capacity.

[e] Dimensional measurements have little value for these magnesium alloys because their internal change, if any, was overshadowed by the surface condition of the specimens. Where the surface corrosion products adhered to the specimens, the dimensional change measurements were quite appreciable. Where these corrosion products had eroded away, the measured dimensional changes were of lesser magnitude.

[f] Izod impact strength.

that Task Groups AZ and BZ are making progress with the design of the test casting die for hot- or cold-chamber casting of zinc alloys.

Task Group E on Pressure Studies has developed a satisfactory method for measuring metal pressures within the test casting die. With this procedure it is planned to study the effect of pressure on the quality of die castings.

The subcommittee is sponsoring a paper by E. N. Jacobi on "Flow Calculations for Die Casting Applied to the A.S.T.M. Committee B–6 Test Casting Die" for publication in the ASTM BULLETIN.[6]

CODE SYSTEM FOR ALLOYS

In view of a number of negative letter ballot votes on the proposed usage of a new code identification system, commonly known as the Frankford Arsenal system, for all of the alloys under the jurisdiction of Committee B-6, the committee has not yet approved its adoption. In order to bring about greater unity on this subject, a Task Group has been appointed to study the negative votes and try to devise a satisfactory compromise, with the idea of later recommending a new letter ballot.

A Nominating Committee, consisting of D. L. Colwell, F. C. Bennett and J. C. Fox, was appointed to present, at the June meeting, nominees for the offices of chairman, vice-chairman, and secretary. The current officers have been reelected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 92 members; 85 members returned their ballots, of whom 78 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

G. L. WERLEY,
Secretary.

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[6] ASTM BULLETIN, No. 166, May, 1950, p. 65.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT[1]

Committee B-7 on Light Metals and Alloys, Cast and Wrought, held two meetings in the past year: one in Atlantic City on June 28, 1949, and the other in Pittsburgh on March 2, 1950.

During the year 44 members were added to the committee and there were 3 removals. At the present time, the committee consists of 76 members of whom; 36 are classified as producers, 28 as consumers, and 12 as general interest members.

The election of offices for the ensuing term of two years resulted in the selection of the following:

Chairman, I. V. Williams.
Vice-chairman, W. Bonsack.
Secretary, J. J. Bowman.

Committee B-7 is continuing to work on the preparation of a corrosion test program under a special subcommittee which was organized last year. This subcommittee has on hand most of the material which is to be exposed during this test; it is expected that the actual exposure of the specimens will start either this year or early next year.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Measuring Thickness of Anodic Coatings on Aluminum by Means of the Filmeter (B 244 – 49 T).

Revision of Tentative Specifications for:

Aluminum-Base Alloy Permanent Mold Castings (B 108 – 48 T), and
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 48 T).

These recommendations were accepted by the Standards Committee on September 22, 1949, and the new and revised tentatives appear in the 1949 Book of A.S.T.M. Standards, Part 2.

Revisions of the Tentative Specifications for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings and Permanent Mold Castings (B 93 – 49 T) were accepted by the Standards Committee on December 14, 1949. The revised tentative specifications appear in the 1949 Book of A.S.T.M. Standards, Part 2.

On December 21, 1949, the Administrative Committee on Standards accepted the following recommendations submitted by Committee B-7:

Revision of Tentative Specifications for:

Magnesium-Base Alloy Sheets (B 90 – 49 T),
Magnesium-Base Alloy Bars, Rods and Shapes (B 107 – 49 T),
Magnesium-Base Alloy Permanent Mold Castings (B 199 – 47 T), and
Magnesium-Base Alloy Extruded Round Tubing (B 217 – 48 T).

The revised tentative specifications

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.**

appear in the 1949 Book of A.S.T.M. Standards, Part 2.

NEW TENTATIVE

The committee recommends the acceptance of the proposed Tentative Specification for Aluminum Alloy Die Forgings as appended hereto.[2]

REVISION OF TENTATIVES

The committee is submitting revisions in 9 tentative specifications. The tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

During the year, the committee has editorially revised all of the product specifications under its jurisdiction to incorporate a revised designation system for the alloys. This designation system is very similar to the one that has been in use for the magnesium alloys, with some revisions which have made it more universally applicable.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specification for Aluminum Bars for Electrical Purposes (B 236 - 48 T) be continued in its present status without change.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to a letter ballot of the committee, which consists of 76 members; 65 members returned their ballots, of whom 57 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

J. J. BOWMAN,
Secretary.

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[2] The new specification was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 2.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Aluminum-Base Alloy Sand Castings (B 26 - 50 T),
Aluminum-Base Alloy Permanent Mold Castings (B 108 - 50 T),
Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 - 50 T),
Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the revised tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 2.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering light metals and alloys which are referred to earlier in this report. The standards appear in their present form in the 1949 Book of A.S.T.M. Standards, Part 2.

REVISIONS OF TENTATIVE SPECIFICATIONS

The committee recommends that the following 9 tentative specifications be revised as indicated below and continued as tentative.

Specifications for Aluminum-Base Alloy Sand Castings (B 26 - 48 T):

Table I.—Add four new alloys and revise the chemical composition of alloy ZG61A as follows:

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Nickel, per cent	Other Elements, per cent	
											Each	Total
ZG61A...	Remainder	0.3	1.0	0.25	0.3	0.5 to 0.65	5.2 to 6.0	0.4 to 0.6	0.10 to 0.25	..	0.05	0.2
ZC81A....	Remainder	0.4 to 1.0	1.0	0.3	0.6	0.2 to 0.5	7.0 to 8.0	0.3	0.2	0.1	0.1	0.2
ZG61B...	Remainder	0.35 to 0.65	0.5	0.15	0.05	0.6 to 0.8	6.0 to 7.0	.....	0.2	...	0.05	0.15
ZG32A...	Remainder	0.2	0.8	0.2	0.4 to 0.6	1.4 to 1.8	2.7 to 3.3	0.2 to 0.4	0.2	...	0.05	...
ZG42A...	Remainder	0.2	0.8	0.2	0.4 to 0.6	1.8 to 2.4	4.0 to 4.5	0.2 to 0.4	0.2	...	0.05	...

Table II.—Add four new alloys and revise the tensile requirements of alloy ZG61A as follows:

Alloy	Condition	Yield[a] Strength, min., psi.	Tensile Strength, min., psi.	Elongation in 2 in., min., per cent
ZG61A..............	T5[b]	22 000	32 000	3.0
ZC81A..............	T5[b]	22 000	30 000	3.0
ZG61B..............	T5[c]	20 000	32 000	2.0
ZG32A..............	T5[b]	17 000	30 000	5.0
ZG42A..............	T5[b]	22 000	33 000	2.0

[a] Yield strength to be determined only when specified in the contract or purchase order.

[b] Aged 21 days at room temperature or artificially aged according to schedule in Table III.

[c] Aged 21 days at room temperature.

Section 6.—Revise to read as follows:

6. (*a*) The tension test specimens representing the castings shall conform to the requirement as to tensile properties shown in Table II. Yield strength tests are to be made on alloys ZG61A, ZC81A, ZG61B, ZG32A, and ZG42A, only when specifically indicated in the contract or purchase order.

(*b*) Although alloys ZG61A, ZC81A, ZG32A and ZG42A are most frequently used in the naturally aged condition, by agreement of the producer and purchaser, the castings may be artificially aged. The producer and purchaser may also agree to base the acceptance of castings on artificially aged test bars. The preferred conditions of artificial aging shown in Table III shall be employed unless other conditions are accepted by mutual consent.

TABLE III.—CONDITIONS FOR ARTIFICIAL AGING

Alloy	Aging
ZG61A.......	356 ± 5 F. for 10 hr.
ZC81A.......	1 day at room temperature +250 ± 10 F. for 16 hr., or 1 day at room temperature +310 ± 5 F. for 9 hr.
ZG32A.......	210 ± 10 F. for 8 hr.
ZG42A.......	210 ± 10 F. for 8 hr.

Section 7 (a).—Revise to read as follows:

7. (*a*) The tension test specimens shall be separately cast in green sand and shall be cast to size according to the dimensions shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) except that the radii at the ends of the reduced section shall be not less than ½ in. They shall not be machined prior to test except to adapt the grip ends to the holders of the testing machine in such a manner as to assure axial loading.

Section 7 (b).—Delete the second sentence.

Specification for Aluminum-Base Alloys for Permanent Mold Castings (B 108 – 49 T):

Table I.—Add data for four new alloys as follows:

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Nickel, per cent	Other Elements, per cent	
											Each	Total
ZC81B......	Remainder	0.4 to 1.0	1.3	0.3	0.6	0.2 to 0.5	7.0 to 8.0	0.3	0.2	0.1	0.1	0.2
ZC60A......	Remainder	0.35 to 0.65	1.4	0.3	0.05	0.25 to 0.45	6.0 to 7.0	......	0.2	...	0.05	0.15
ZG32A......	Remainder	0.2	0.8	0.2	0.4 to 0.6	1.4 to 1.8	2.7 to 3.3	0.2 to 0.4	0.2	...	0.05	...
ZG42A......	Remainder	0.2	0.8	0.2	0.4 to 0.6	1.8 to 2.4	4.0 to 4.5	0.2 to 0.4	0.2	...	0.05	...

Table II.—Add data for four new alloys as follows:

Alloy	Condition	Yield Strength,[a] min., psi.	Tensile Strength, min., psi.	Elongation in 2 in. min., per cent
ZC81B..............	T5[b]	22 000	32 000	4
ZC60A..............	T5[c]	....	28 000	7
ZG32A..............	T5[b]	17 000	37 000	10
ZG42A..............	T5[b]	25 000	42 000	4

[a] Yield strength to be evaluated only when specified in contract or purchase order.

[b] Aged 21 days at room temperature or artificially aged as shown in Table III.

[c] Aged 21 days at room temperature.

Section 6.—Revise to read as follows:

6. (*a*) The tension test specimen representing the castings shall conform to the requirement as to tensile properties shown in Table II. Yield strength tests are to be made on alloys ZC81B, ZG32A and ZG42A only when specifically indicated in the contract or purchase order.

(*b*) Although alloys ZC81B, ZG32A and ZG42A are most frequently used in the naturally aged condition, by agreement of the producer and purchaser, the castings may be artificially aged. The producer and purchaser may also agree to base the acceptance of castings on artificially aged test bars. The preferred conditions of artificial aging shown in Table III shall be employed unless other conditions are accepted by mutual consent.

TABLE III.—AGING CONDITIONS.

Alloy	Aging
ZC81B.......	1 day at room temperature +250 ± 10F for 16 hr.
ZG32A.......	210 F. for 8 hr.
ZG42A.......	210 F. for 8 hr.

Section 7 (a).—Revise to read as follows:

7. (*a*) The tension test specimens shall be separately cast in permanent molds and shall be cast to size in accordance with Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),[1] except that the radii at the ends of the reduced section shall be not less than ½ in. They shall not be machined prior to test except to adapt the grip ends to the holders of the testing machine in such a manner as to assure axial loading.

Section 7 (b).—Delete the second sentence.

Specifications for Aluminum and Aluminum-Alloy Sheet and Plate for Use in Pressure Vessels (B 178 – 49 T):

Table I.—Add requirements for alloys MG11A and G1A as follows:

	MG11A	G1A
Copper, per cent	0.20	0.25
Iron, per cent	0.70	0.80
Silicon, per cent	0.30	0.50
Manganese, per cent	1.0 to 1.5	0.15
Magnesium, per cent	0.8 to 1.3	1.0 to 1.8
Zinc, per cent	0.10	0.25
Chromium, per cent	....	0.10
Titanium, per cent	....	....
Other Elements, per cent, Each	0.05	0.05
Other Elements, per cent, Total	0.15	0.15
Aluminum, per cent	Remainder	Remainder

Table II.—Add requirements for alloys MG11A and G1A as follows:

Temper	Thickness, in.	Tensile Strength, min., psi.	Elongation in 2 in., min., per cent
	ALLOY MG11A		
O	0.051 to 0.249	23 000	18
	0.250 to 3.000	23 000	16
H32	0.051 to 0.113	28 000	5
	0.114 to 0.249	28 000	6
	0.250 to 2.000	28 000	6
H34	0.051 to 0.113	32 000	4
	0.114 to 0.249	32 000	5
	0.250 to 1.000	32 000	5
H112	0.250 to 3.000	23 000	7
	ALLOY G1A		
O	0.051 to 0.113	19 000	20
	0.114 to 0.249	18 000	22
	0.250 to 1.000	18 000	20
H32	0.051 to 0.200	22 000	6
H34	0.051 to 0.140	25 000	5
H112	0.201 to 1.000	20 000	12

[d] Not required.

Section 7.—Revise to read as follows:

7. Tension test specimens for sheet or plate in the H12, H32, H14 and H34 tempers shall be taken parallel to the direction of rolling; for sheet or plate in all other tempers, the tension test specimens may be taken in any direction (Note). For sheet and plate less than 0.500-in. in thickness, the standard tension test specimen for sheet metal, as shown in Fig. 2 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),[1] shall be used. For plate 0.500 in. or over in thickness, the standard ½-in. diameter specimen shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),[1] or a similar round specimen having the maximum possible dimensions proportional to those of the standard ½-in. diameter specimen, shall be used. For plate over 1½ in. in thickness, the specimen shall be taken midway between the center and surface of the plate. (Retain note).

Table III.—Revise title of Table III to include alloy G1A.

Table IV.—Revise title of Table IV to include alloy MG11A.

Appendix.—Add the following data for alloys MG11A and G1A:

Temper	Thickness, in.	Yield Strength, psi.
	ALLOY MG11A	
O	0.051 to 3.000	8 500
H32	0.051 to 2.000	21 000
H34	0.051 to 1.000	25 000
H112	0.250 to 3.000	9 000
	ALLOY G1A	
O	0.051 to 1.000	6 000
H32	0.051 to 0.200	17 000
H34	0.051 to 0.140	21 000
H112	0.201 to 1.000	10 000

Specification for Aluminum Base Alloys in Ingot Form For Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 49 T):

Table I.—Add five new alloys with

the following chemical composition requirements:

Alloy	Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Nickel, per cent	Other Elements, per cent	
											Each	Total
ZC81A-B.....	Remainder	0.4 to 1.0	0.9	0.25	0.6	0.2 to 0.5	7.0 to 8.0	0.3	0.2	0.1	0.05	0.2
ZG32A.......	Remainder	0.2	0.6	0.2	0.4 to 0.6	1.4 to 1.8	2.7 to 3.3	0.2 to 0.4	0.2	...	0.05	...
ZG42A.......	Remainder	0.2	0.6	0.2	0.4 to 0.6	1.8 to 2.4	4.0 to 4.5	0.2 to 0.4	0.2	...	0.05	...
ZG61B.......	Remainder	0.35 to 0.65	0.4	0.15	0.05	0.6 to 0.8	6.0 to 7.0	...	0.2	...	0.05	0.15
ZC60A.......	Remainder	0.35 to 0.65	1.3	0.3	0.05	0.25 to 0.45	6.0 to 7.0	...	0.2	...	0.05	0.15

Table III.—Revise title of Table III to include reference to alloy G1A.

Specifications for Aluminum and Aluminum-Alloy Sheet and Plate (B 209 – 49 T):

Table I.—Add requirements for alloy G1A, using chemical composition limits given above in revised Table I of B 178 – 49 T.

Table II.—Add physical requirements for alloy G1A as follows:

Temper	Thickness, in.	Tensile Strength, psi.	Yield Strength, min., psi.	Elongation in 2 in., min., per cent	Bend
ALLOY G1A					
O......	0.016 to 0.019	24 000 max.	..	16	0[c]
	0.020 to 0.031	24 000 max.	..	18	0[c]
	0.032 to 0.113	24 000 max.	..	20	0[c]
	0.114 to 0.249	24 000 max.	..	22	0[c]
	0.250 to 1.000	24 000 max.	..	20	2
H32....	0.020 to 0.050	22 000 min.	..	4	1
	0.051 to 0.200	22 000 min.	..	6	2
H34....	0.016 to 0.031	25 000 min.	..	3	1
	0.032 to 0.050	25 000 min.	..	4	1
	0.051 to 0.140	25 000 min.	..	5	2
H36....	0.016 to 0.019	27 000 min.	..	2	3
	0.020 to 0.050	27 000 min.	..	3	3
	0.051 to 0.102	27 000 min.	..	4	4
H38....	0.016 to 0.031	29 000 min.	..	2	[d]
	0.032 to 0.050	29 000 min.	..	3	[d]
	0.051 to 0.072	29 000 min.	..	4	[d]
F......	0.016 to 1.000	[d]	..	[d]	[d]

[d] Not required.

Section 8 (a).—Change to read as above for revised Section 7 of B 178 – 49 T.

Tentative Specifications for Aluminum-Alloy Drawn Seamless Tubes (B 210 – 49 T):

Table I.—Add chemical composition requirements for alloy MG11A given above in revised Table I of B 178 – 49 T.

Table II.—Add tensile requirements for alloy MG11A in annealed temper only. These are 29,000 psi. max., tensile strength for wall thicknesses 0.018 to 0.450 in. No requirements for yield strength or elongation.

Section 7.—Revise to read as follows:

> 7. Tension test specimens from round tubes 2 in. or less in outside diameter and from square tubes 1½ in. or less on a side shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a specimen. Specimens of the types shown in Figs. 3 or 11 (specimen No. 1) of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8)[1] may be used for round tubes over 2 in. in diameter, for square tubes over 1½ in. on a side, for all sizes of tubes other than round and square, or in those cases when a full-section specimen can not be used; small size specimens with dimensions proportional to those shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8)[1] may be used when necessary.

Specification for Aluminum-Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 – 48 T):

Table I.—Revise to permit 0.10 per cent max. magnesium in the coating of alloy M1A Clad Tubes and to change

Footnote "*a*" under Table I by replacing 0.60 per cent with 0.70 per cent.

Section 7.—Revise to read as follows:

> 7. Tension test specimens shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a specimen. Specimens of the type shown in Fig. 11 (specimen No. 1) of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) may be used in those cases when a full-section specimen can not be used.

Specifications for Aluminum-Alloy Extruded Tubes (B 235 – 49 T):

Section 7.—Change to read as indicated above for revised Section 7 of B 210 – 49 T.

Table VI.—Revise Table VI by deleting the tabular form and substituting the following statement:

> "Permissible variations of wall thickness at any point from specified wall thickness shall not be greater than plus or minus 10 per cent of the specified wall thickness: maximum 0.060 in., minimum 0.010 in."

Specifications for Aluminum-Alloy Pipe (B 241 – 49 T):

Table I.—Change the limit for iron in alloy GS10A from 0.35 per cent to 0.50 per cent.

Section 7.—Revise to read as follows:

> 7. Tension test specimens of pipes $1\frac{1}{2}$ in. or less in size shall be a full section of the pipe unless the limitations of the testing machine preclude the use of such a specimen. For pipes in sizes greater than $1\frac{1}{2}$ in., or in those cases when a full-section specimen can not be used it is permissible to use specimens of the type shown in Fig. 11 (specimen No. 1) of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8)[1] or small size specimens with dimensions proportional to those shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8).[1]

EDITORIAL CHANGES IN TENTATIVES

When the following tentatives are next revised for other reasons, the changes outlined below are recommended as editorial:

Specifications for Magnesium-Base Alloy Sand Castings (B 80 – 49 T):

Section 8 (a).—Revise as indicated for Section 7 (*a*) of Tentative Specifications B 26.

Specifications for Magnesium-Base Alloy Sheet (B 90 – 49 T):

Section 7.—Revise to read as follows:

> 7. The tension test specimens shall be cut in the direction of rolling or longitudinal direction and shall be machined to the form and dimensions shown in Fig. 2 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8).

Specifications for Magnesium-Base Alloy Forgings (B 91 – 49 T):

Section 7.—Revise to read as follows:

> 7. Tension test specimens shall be taken from a forging or from a separately forged coupon made from material representative of the forgings, in such a manner that the longitudinal axis of the specimen is parallel to the direction of maximum flow of the metal in the forging. The specimens shall be machined to the form and dimensions shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8), or in the case of thin material may be machined to the form and dimensions shown in Fig. 2 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8).

Specifications for Magnesium-Base Alloy Bars, Rods and Shapes (B 107 – 49 T):

Section 7 (a).—Revise to read as follows:

> 7. (*a*) Tension test specimens shall be a full section of the material or of the type shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8). Small-size specimens with dimensions proportional to those shown in Fig. 3 (of E 8), or a sheet tension specimen of the type shown in Fig. 2 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),[1] may be used if the size or shape of the material makes it impracti-

cable to use a full-section or the standard round specimen.

Specifications for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 49 T):

Section 8 (a).—Revise to read as follows:

8. (*a*) The tension test specimens shall be separately cast in permanent molds and shall be cast to size in accordance with Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8),[1] except that the radii at the ends of the reduced section shall be not less than ½ in. They shall not be machined prior to test except to adapt the grip ends to the holders of the testing machine in such a manner as to assure axial loading.

Specifications for Aluminum and Aluminum-Alloy Bars, Rods and Wire (B 211 – 49 T):

Section 7 (a).—Change to read as indicated above for revised Section 7 (*a*) of B 107 – 49 T.

Specifications for Magnesium-Base Alloy Extruded Round Tubes (B 217 – 49 T):

Section 7.—Revise to read as follows:

7. Tension test specimens from tubes 2 in. or less in outside diameter shall be of the full section of the tube unless the limitations of the testing machine preclude the use of such a specimen. For tubes over 2 in. in diameter, or in those cases when a full-section specimen can not be used it is permissible to use specimens of the type shown in Fig. 11 (specimen No. 1) of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8)[1] or small size specimens with dimensions proportional to those shown in Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation : E 8).[1]

Specifications for Aluminum and Aluminum-Alloy Extruded Bars, Rods and Shapes (B 221 – 49 T):

Section 7 (a).—Change to read as indicated above for revised Section 7 (*a*) of B 107 – 49 T.

Specifications for Aluminum Bars for Electrical Purposes (Bus Bars) (B 236 – 48 T):

Section 9—Change first two sentences to read as indicated above for Section 7(*a*) of Specifications B 107. Retain last sentence.

REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS[1]

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: one on October 14, 1949, in Chicago, Ill., in conjunction with the Fall Congress of the Electrochemical Society, and one in Pittsburgh, Pa., on March 4, 1950.

The membership of the committee now totals 138 members, of whom 44 are classified as producers, 22 as consumers, 66 as general interest, and 6 as consulting members.

The following officers were elected for the ensuing term of two years:

Chairman, C. H. Sample.

Vice-Chairman, K. G. Soderberg.

Secretary, R. B. Saltonstall.

Advisory Committee Member-at-large, B. C. Case.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends that the following tentatives be continued as tentative without revision:

Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (**A 166 - 45 T**).—Committee B-8 recommends continuation as tentative, pending the outcome of exposure tests presently being conducted by Subcommittee II.

Methods of Test for Local Thickness of Electrodeposited Coatings (**A 219 - 45 T**).—The committee recommends continuation as tentative in view of the fact that the reproducibility of the methods of test covered by the specification is currently being investigated by Section A of Subcommittee III.

Specifications for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-base Alloys (**B 142 - 45 T**).—The committee recommends continuation as tentative, pending the outcome of large scale private tests now in progress.

Specifications for Electrodeposited Coatings of Lead on Steel (**B 200 - 45 T**).—The committee recommends continuation as tentative, pending a study of the results of exposure tests conducted by Subcommittee II.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications, Papers, and Definitions (J. E. Stareck, chairman).—A section of this subcommittee (K. G. Soderberg, chairman) has drafted modifications of Specifications A 164, A 165, and A 166 to point out that they are not applicable to many threaded components because of difficulties with dimensional tolerances.

The same section has drafted modifications of Specifications A 164 and A 165 to point out the danger of making recommendations for atmospheric exposure from salt spray (fog) test results.

Another active section of subcommittee I (M. M. Beckwith, chairman) has canvassed available information on the effect of thickness of chromium on thin nickel coatings over copper alloys, with respect to stress cracking of the nickel deposits. Existing information is not sufficiently conclusive to be a basis for changes in present specifications for chromium thickness. Exposure tests to yield such information are suggested; also a footnote to Specifications B 141 suggesting agreement between purchaser and manufacturer on lower minimum thicknesses of chromium in certain cases.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

Subcommittee I is considering, through a working section, (F. Nagley, chairman), the desirability of including metric as well as English units in specifications under the jurisdiction of Committee B-8; also the question of significant figures.

Subcommittee II on Performance Tests (H. A. Pray, chairman).—This subcommittee has continued exposure tests of electrodeposited lead coatings on steel, and copper-nickel-chromium on high-carbon steel and is continuing the periodic removal of lead-coated steel panels for steel weight loss and tension tests.

Supplementary panels have been added to the copper-nickel-chromium group including chromium and nickel-chromium deposits on rolled nickel sheet, and additional copper-nickel-chromium deposits on high-carbon steel, the latter for the purpose of further investigating the effect of copper.

Progress is also being made in developing photographic standards to aid in inspecting the panels.

Salt spray (fog) tests have been performed on a set of the panels and results will be included in the final report.

Subcommittee III on Conformance Tests. (W. A. Wesley, chairman).—The resignation of Chairman Wesley, effective June 1, 1950, has been accepted with regret, and B. C. Case has been appointed to succeed him.

Four working sections of this subcommittee have been active throughout the year.

Section A on Thickness Tests (A. H. DuRose, chairman) has a program in progress to investigate the reproducibility of various thickness test methods, that is, magnetic, stripping, microscopic and dropping tests. A number of specimens have been prepared and distributed to cooperating laboratories for magnetic thickness measurements. The data are being analyzed statistically, and the other methods of thickness measurement will be investigated in a similar manner on the same specimens.

Section B on Porosity Tests (A. Mendizza, chairman), through the cooperation of several firms, has had a number of steel panels, with indentations formed by the Olsen Cup Method, nickel plated for salt spray (fog) test.

Section C on Mechanical Property Tests (M. M. Beckwith, chairman), has been measuring the hardness of electrodeposited coatings by the Knoop Indentor Method. No apparatus or method for adhesion testing has been found which appears to warrant testing by the Section. Information on promising ductility test methods is being obtained.

Section D on Luster Tests (G. Jernstedt, chairman), has investigated quantitative methods of measuring luster such as reflectivity and surface analysis. It appears that reflectivity measurements alone are not sufficient to define luster, and that surface analyzer readings must be included.

Subcommittee IV on Plating Practice (K. M. Huston, chairman).—This subcommittee has been very active through its several working sections which have either prepared, or have in the course of preparation, Recommended Practices as follows:

Section B (E. A. Anderson, chairman), "Recommended Practice for the Preparation of Zinc Base Die Castings for Plating."

Section C (E. S. Coe, chairman), "Recommended Practice for the Preparation of Copper and Copper Alloys for Electroplating."

Section D (K. M. Huston, chairman), "Recommended Practice for the Preparation of and Plating on Stainless Steel."

Section E (F. Keller, Chairman), "Recommended Practice for the Preparation of Aluminum for Electroplating."

Section F (H. Narcus, chairman), "Recommended Practice for Preparation of Plastics for Electroplating."

Authorization has been given for the appointment of two new sections to investigate the desirability of preparing recommended practices for plating (*1*) on lead and tin and their alloys and (*2*) on cast and malleable iron.

Subcommittee V on Supplemental Treatments of Metallic Coatings (Marc Darrin, chairman).—Sections B on Accelerated Tests (A. Mendizza, chairman), C on Performance Tests (R. E. Harr, chairman), and D on Surface Treatments for Cadmium (L. O. Gilbert, chairman) have been working in close cooperation. An exploratory outdoor exposure study of chromate-treated zinc and cadmium finishes has been completed, and plans have been formulated for a more extensive program to investigate five types of chromate treatments which will include the exposure of treated specimens to (*a*) industrial atmosphere, (*b*) marine atmosphere, and (*c*) warehouse storage and to study accelerated corrosion in (*a*) 100 per cent humidity, (*b*) pressure cooker, (*c*) salt spray (fog), and (*d*) water immersion. The effect of the hexavalent chromium content of the surface films will also be investigated.

Section E on Phosphate Treatments (A. L. Alexander, temporary chairman) has under way an experimental program to develop possible methods for evaluating the effectiveness of phosphate treatments for corrosion protection and as a surface preparation for subsequently applied finishes.

This report has been submitted to letter ballot of the committee, which consists of 128 voting members; 92 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

R. B. SALTONSTALL,
Secretary.

REPORT OF COMMITTEE B-9
ON
METAL POWDERS AND METAL POWDER PRODUCTS[1]

This report covers the activities of Committee B-9 on Metal Powders and Metal Powder Products for the period from June, 1949, to June, 1950. During this time, one meeting was held in New York, N. Y., on February 13, 1950.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. A. Reich.
Vice-Chairman, D. S. Urquhart.
Secretary, A. Squire.

Additional members appointed to the Advisory Committee are as follows:

J. E. Drapeau, Jr.,
F. P. Peters,
R. P. Seelig,
J. F. Kuzmick.

The committee notes with sadness the death of Carter S. Cole of the A.S.T.M. Headquarters Staff. The membership of Committee B-9 particularly benefited from his kind advice and assistance in carrying out matters of both internal interest and general A.S.T.M. procedure.

REVISION OF TENTATIVE

Tentative Specifications for Sintered Metal Powder Structural Parts (**B 222 – 47 T**).[2]—The committee recommends an addition to the scope of these specifications as follows:

Section 1.—Add the following sentence: "These parts contain a controlled amount of porosity which may be utilized for oil impregnation."

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF ADVISORY COMMITTEE

The Advisory Committee held two meetings during the year to act on various problems connected with the administration of the committee. Some success has been attained in enlarging the consumer representation on Committee B-9.

In addition, a meeting was held of representatives of A.S.T.M. Headquarters, Committee B-9, and officers of the Metal Powder Association to discuss methods of coordinating parallel activities of the two agencies in the field of test methods and specifications. A tentative arrangement of cross-referencing of standards was drawn up, subject to the approval of the two groups.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman). —The glossary of terms used in powder metallurgy drawn up by this subcommittee has been published as the Tentative Definitions of Terms Used in Powder Metallurgy (B 243 – 49 T).[2] It has been decided that a lead paragraph should be added to this glossary explaining that the glossary is composed

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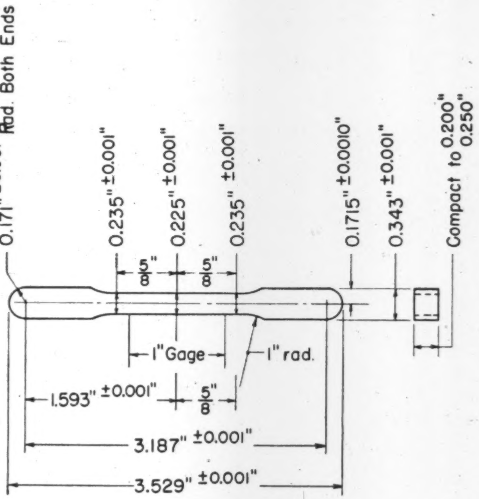
[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 2.

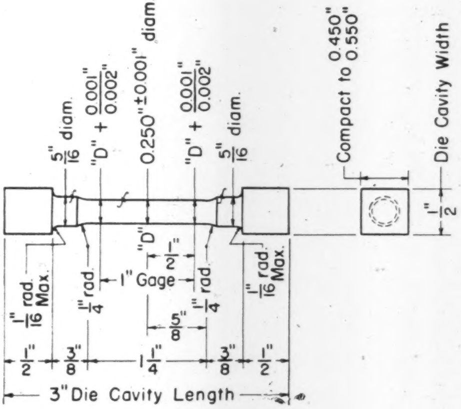
[3] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.


0.171" ±0.001" Rad. Both Ends
0.235" ±0.001"
0.225" ±0.001"
0.235" ±0.001"
0.1715" ±0.0010"
0.343" ±0.001"
Compact to 0.200" 0.250"
5/8"
5/8"
1" Gage
1" rad.
1.593" ±0.001"
5/8"
3.187" ±0.001"
3.529" ±0.001"

Pressing Area = 1.00 sq. in.

Dimensions Specified Are Those of
The Compact When in The Die.

Fig. 1.—Flat Unmachined Tension Test Bar for Powdered Metals.


5/16" diam.
"D" + 0.001"/0.002"
0.250" ±0.001" diam.
"D" + 0.001"/0.002"
5/16" diam.
Compact to 0.450" 0.550"
Die Cavity Width
1/2"
"D"
1/2"
1" Gage
1/16" rad. Max.
1/4" rad.
1/4" rad.
1/16" rad. Max.
5/8"
1/2"
3/8"
1 1/4"
3/8"
1/2"
3" Die Cavity Length

Pressing Area of Unmachined Compact = 1.5 sq. in.

Machining Recomendations

1. Rough Machine to 5/16" diam.
2. Finish Turn 0.250" diam. with Radii and Taper.
3. Polish with 00 Emery Cloth.
4. Lap with Crocus Cloth.

Fig. 2.—Round Machined Tension Test Bar for Powdered Metals

only of terms not generally used in the same sense in fields other than powder metallurgy. This paragraph is being drafted for submission to main committee ballot during the coming year.

Subcommittee I has agreed on the design of two standard tension test bars for use in powder metallurgy work. One of these designs is for a bar which is to be used in the pressed and sintered condition while the other is to be machined. These designs, Figs. 1 and 2, have been submitted to main committee ballot, the results of which will be reported at the Annual Meeting.

Subcommittee II on Metal Powders (D. O. Noel, chairman).—One meeting of this subcommittee was held during the year. The following tests methods are being written in A.S.T.M. form for ballot by the members and subsequent submittal to Committee E-3:

1. Test for "Hydrogen Loss" in metal powders.
2. Test for insoluble matter in iron and copper powders.
3. Test for free and total iron in iron powder.

In addition, a method for subsieve particle analysis using the Roller Air Analyzer is being drafted in final form for approval by the subcommittee. Additional work on the compressibility of metal powders has resulted in a proposed method which will be tried by several cooperating laboratories prior to submittal to committee ballot.

A review of work on subsieve particle analysis using the Cenco Photolometer is being made by a special subgroup. Methods of determining green strength and green density of metal powder compacts are also under consideration.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman) Section A on Bearings (D. S. Urquhart, chairman) engaged in the following activities during the year: Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 45 T) were discussed with a view to changing the range of carbon content to 1.75 per cent, maximum, for classes A and B of type I material. This change was voted by the subsection and is to be submitted to main committee ballot. A proposed addition to Specifications B 202 giving recommended press fits and running clearances was tentatively agreed upon and is being drawn up for ballot. The section also considered an addition to Specifications B 202 to cover a new class of type II material having a low carbon content and is continuing its efforts to develop some kind of standardization of bearing sizes which will be useful to producers and consumers of metal powder bronze and iron bearings.

Subcommittee III-B on Structural Parts (F. V. Lenel, chairman).—This group held one meeting during the year and the following action was taken: The change in scope of Specifications B 222 – 47 T, noted earlier in this report, was recommended. This change recognizes the upper limit in density carried in the specifications and the usefulness of the self-lubricating property thereby imparted, in some applications.

The study group on copper-tin alloy structural parts reported that specifications covering 90 per cent copper, 10 per cent tin alloy should be drafted to include two density ranges, 6.4 to 6.8 g. per cu. cm. and 6.8 to 7.2 g. per cu. cm. Such specifications are in the process of being drafted by the study group. Manufacturers of the 95 per cent copper, 5 per cent tin structural alloy are being canvassed for proposed specifications covering this material to be considered by the group.

Work is being continued on a draft of specifications for three brass parts made from metal powders.

With reference to copper impregnated iron and steel parts, a draft of specifications on this material is being prepared for consideration by the group.

Section C on Cemented Carbides (J. C. Redmond, chairman) met twice during the year and made some progress in the preparation of several standards and test methods.

A task group on metallographic preparation has conducted an extensive survey of the variation in interpretation and classification of carbides according to their porosity. This survey indicated substantial agreement in interpretation of results but a second survey is being made before a definite method of classification is proposed for adoption.

A subgroup appointed to study the transverse rupture test as a quality control method reported that the results of this test are so dependent upon specimen size and preparation that it is not recommended as an acceptance test. A second group on hardness testing of carbides met during the year, but no specific test methods were agreed upon. This group was reconstituted to make a further study of the problem.

Work was done in an effort to prepare a usable chart of cemented carbide grade designations. This chart has been considered carefully and returned to the subgroup for further consideration.

Section D on Friction Materials (F. F. White, chairman) held no meetings during the year but considerable progress was made towards the development and standardization of a machine for testing the frictional characteristics of metal powder materials. As soon as sufficient data to show that this machine is satisfactory have been accumulated, its design will be recommended for use in testing friction facings.

This report has been submitted to letter ballot of the committee, which consists of 50 members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. REICH,
Chairman.

A. SQUIRE,
Secretary.

REPORT OF JOINT COMMITTEE
ON
EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS[1]

The ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals met on December 2, 1949, during the Annual Meeting of the American Society of Mechanical Engineers in New York. This meeting was preceded by a meeting of the Technical Advisory Committee. Constructive reports indicating much progress in the work were received from the several panels and project committees.

In a technical session sponsored jointly by the Gas-Turbine Power Division and the Joint Committee, two papers were presented:

"The Outlook for Ceramics in Gas Turbines" by I. E. Campbell and W. H. Duckworth,
"Metallurgy of Gas-Turbine Materials" by N. L. Mochel.

In another session cosponsored with the Research Committee on High-Temperature Steam Generation, two papers were presented:

"Heat Transfer to Superheated Steam at High Pressures" by W. H. McAdams, W. E. Kennell, and J. N. Adams,
"Possibilities of the Regenerative Steam Cycle at Temperatures up to 1600 F." by P. H. Knowlton and R. W. Hartwell.

Funds in the hands of the ASME to the credit of the committee amounted to $8698.40 as of May 31, 1950. Of these funds, the sum of $3500 is earmarked to cover costs of Project 18 at Battelle Memorial Inst.; $1000 is earmarked for the creep data surveys, to cover costs at Battelle for preparing and circulating a transmittal letter and data questionnaire to possible cooperating laboratories, and another $1000 is to be allocated for work on non-ferrous metals. The questionnaire requests data for use in a revised compilation on cast and wrought austenitic steels.

An important publication was issued under the auspices of the Joint Committee, dated March, 1950, constituting a "Report on the Strength of Wrought Steels at Elevated Temperatures," prepared by R. F. Miller and J. J. Heger, Carnegie-Illinois Steel Corp. This 116-page publication, $8\frac{1}{2}$ by 11-in. page size, contains a great amount of data, largely in graphical form, on both plain carbon and alloy steels, the latter including ferritic and austenitic steels.

An important new Subcommittee on Low-Temperature Problems has been organized, and held its first meeting in April, this group functioning under the Petroleum and Chemical Panel.

Preceded by meetings of most of the Panels and the Technical Advisory Committee the main committee met on Tuesday, June 27, during the Annual Meeting of the ASTM at Atlantic City. Reports were received from the Panel chairmen and from Projects 18 and 29.

The Joint Committee is sponsoring two symposiums and a round-table discussion at this Annual Meeting. The Gas-Turbine Panel arranged for a Symposium on Corrosion of Materials at Elevated Temperatures[2] and a Round-Table Discussion on Heavy Forgings. The General Research Panel arranged a Symposium on the Nature, Occurrence, and Effects of Sigma Phase.[3] In addition, the Joint Committee sponsored a session on Effect of Temperature on the Properties of Metals at which the following papers were presented:

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] To be issued as a separate technical publication, *STP 108*.

"The Effect of Various Treatments on the Fatigue Strength of Notched S-816 and Timken 16-25-6 at Elevated Temperatures," by W. E. Jones and G. B. Wilkes, Jr., General Electric Co.,[4]

"Hardened Alloy Steel for Service Up to 700 F." by G. V. Smith, W. B. Seens, and E. J. Dulis, U. S. Steel Corp.,[5]

"The Selection of a Limited Number from Many Possible Conditioning Treatments for Alloys to Achieve Best Coverage and Statistical Evaluation," by J. M. Cameron and W. J. Youden, Nat. Bureau of Standards,[6]

"Effect of Rate of Strain on the Flow Stress of Gas Turbine Alloys at 1200 and 1500 F., by M. J. Manjoine, Westinghouse Electric Corp.,[7]

"Chromium-Base Alloys," by W. L. Havekotte, C. T. Greenidge, and H. C. Cross, Battelle Memorial Inst.,[8]

"Hardening of Austenitic Stainless Steels by Mechanical Working at Sub-Zero Temperatures," by N. A. Ziegler and P. H. Brace, Westinghouse Electric Corp.,[9]

"Effect of Temperature on the Mechanical Properties, Characteristics and Processing of Austenitic Stainless Steels," by V. N. Krivobok and A. M. Talbot, International Nickel Co., Inc.[10]

A tentative Recommended Practice for Conducting Time for Rupture Tests of Metallic Materials prepared by a subgroup of the Gas-Turbine Panel, and approved by the main committee, has received the approval of the A.S.T.M. Administrative Committee on Standards and is being printed by the Society, carrying the A.S.T.M. designation E 85 - 50 T.

The work of the committee has expanded greatly and many more individuals are now participating in the activities. In order that there should be clear understanding of the operations of the committee and how it functions, a set of "By-Laws and Information" has been drafted which is now under consideration in the committee.

Notes on the activities of certain of the Panels and Research Projects follow:

Aviation Panel (Leo Schapiro, chairman):

Project AP1A, "Statistical Evaluation of Creep-Rupture Properties of Certain Sheet Materials," is being instructed to proceed with the accumulation of materials. Project AP2A, "Exploratory Investigation of High-Temperature Sheet Materials for Power Plant Applications," has accumulated some of its test materials and has made distribution to cooperating companies. A new project is under discussion relating to elevated temperature properties of aluminum and magnesium alloys, but no action was taken pending the receipt of more information from airframe builders.

Gas Turbine Panel (C. T. Evans, Jr., chairman):

The Panel voted to appoint the chairman as a liaison representative with the Gas Turbine Power Division of ASME. Project GTP1 on fatigue was reviewed with particular reference to the program to determine the effects of notches on high-temperature fatigue. There was discussion on the subject of brittleness and the relation of design factors to brittle behavior. Representatives of material manufacturers and gas turbine design engineers will be invited to attend the next meeting of the Panel for a full discussion of this subject. Sessions are being sponsored by the Gas Turbine Panel at this annual meeting as indicated earlier in the report.

General Research Panel (A. J. Herzig, chairman):

At its fifth meeting, the General Research Panel received its initial progress report on a project sponsored at Westinghouse Research Laboratory on the Effect of Stress Concentration Factors at Elevated Temperatures.

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[3] To be issued as a separate technical publication, *STP 110*.

[4] See p. 744.

[5] See p. 882.

[6] See p. 951.

[7] See p. 931.

[8] See p. 1101.

[9] See p. 861.

[10] See p. 895.

The Panel offered a paper on the subject of Arc-Cast Molybdenum-Base Binary Alloys for the Joint Committee sponsored session at the 1950 Annual Meeting of ASME.

Petroleum and Chemical Panel (C. L. Clark, chairman):

Among the active projects under way in subcommittees of this panel are the following:

Project	Title	Chairman
No. 1	Compilation of Experience with Metals above 1500 F.	H. W. Schmidt
No. 2	Behavior and Application of Metals at Low Temperatures	I. Roberts
No. 3	Relative Merits of 18 Cr-8 Ni versus the Stabilized Grades of Austenitic Steels	M. S. Northup
No. 4	Necessity of Preheating and Stress Relieving Ferritic Steels Welded with Austenitic Rods.	W. L. Bowler
No. 5	The Cracking at the Welds of 12 Cr type liners in Pressure Vessels	M. W. Barnes
No. 6	Differences in Graphitization in the Steam Power Field as Compared to the Petroleum and Chemical Industries	W. B. Hoyt
No. 7	Experiences with Corrosion by Vanadium Compounds	E. F. Tibbetts

A paper is being prepared by M. E. Holmberg on "Experiences in the Petroleum Industry with Austenitic Stainless Steels under Cyclic Heating and Stressing Conditions" for presentation to the ASME meeting in December. During the same meeting E. F. Tibbetts will present a report of Project No. 7 at a symposium to be cosponsored by the Joint Committee with the Fuels Division of ASME.

Low-Temperature Group of the Chemical and Petroleum Panel (Irving Roberts, chairman):

The organization meeting of the Low-Temperature Group was held in Pittsburgh, Pa., on April 24, 1950. The following subjects and questions came up for discussion:

1. What criteria or specifications determine the suitability of materials for low-temperature service?
2. Standardization of physical test procedures, particularly impact testing.
3. Correlation of impact tests with low-temperature failures.
4. Stability of physical properties, and low-temperature shift of certain alloys from austenitic to ferritic.
5. Ability of materials to yield locally.
6. Triaxial stress systems.
7. Effect of steel "history" in the mill.
8. Test requirements vary with the type of material, design, and service conditions.
9. Low-temperature failures.
10. Which of the notch-sensitivity or impact specimens most realistically approaches service conditions in actual structures?
11. How will plants conform to state codes, when there is no specific code for low-temperature service?
12. Fatigue in service.
13. What about correlating data on elastic modulus, thermal conductivity, coefficient of expansion, etc., at low temperatures?

Following extended discussion of these points, the following three subgroups were named:

1.—Test Methods and Procedures
2.—Present Industrial Practice including service failures and physical properties)
3.—Bonding (including welding, brazing and soldering).

It was generally agreed that the Low-Temperature Group confine its activities to industrial applications, and that the most important job of the subgroups is that of abstracting the technical literature.

Liaison Report from ASME Special Research Committee on High-Temperature Steam Generation (C. L. Clark):

The primary function of this Special ASME Research Committee is to ascertain the feasibility of steam generation at temperatures in excess of the maximum now used. Since the hottest section of the superheater represents the extreme condition, in so far as metal temperatures are concerned, the effects of steam and furnace gases on the potentially available alloys will have to be determined before commercial advantage can be taken of the higher operating temperatures.

This Special Committee therefore proposes to determine:

1. The method of formation, thickness, permanence, and thermal conductivity of oxide films on the steam swept surfaces.
2. The resistance of the alloys to the furnace gases such as may be encountered in commercial service, that is, the action on the exterior tube surfaces.
3. The metallurgical stability of the contemplated materials subjected to high temperatures for long periods of time.
4. The effects of rapid tube quenching as experienced during soot blowing.

The actual research program will be undertaken at Purdue University, at Twin Branch Power Plant at Mishawaka, Ind., and at Battelle Memorial Inst.

The undersigned officers of the Joint Committee were reelected for a two-year term ending June, 1952, and, together with A. J. Herzig and N. L. Mochel, constitute the Executive Committee.

Respectfully submitted on behalf of the committee,

ERNEST L. ROBINSON,
Chairman.
F. B. FOLEY,
Vice-Chairman.

H. C. CROSS,
Secretary.

REPORT OF COMMITTEE C-1
ON
CEMENT[1]

Committee C-1 on Cement held three meetings during the past year: in Atlantic City, N. J., July 1, 1949; in San Francisco, Calif., October 11, 1949; and in Pittsburgh, Pa., March 1, 1950.

At the July 1 meeting, F. R. McMillan, formerly Director of Research for the Portland Cement Association, presented an interesting talk on "Present Trends in the Long-Time Study of Cement and Concrete." That study has been conducted by the Portland Cement Association at various places in the country for some years, under the guidance of a special committee of technical representatives of government, states and producer interests, with P. H. Bates as chairman.

The October meeting was the first Pacific Coast meeting of the committee. It was well attended and featured numerous papers, presented under the sponsorship of the committee. Two papers dealt with portland cements, their types, and their performance in long-time observations. In addition a symposium on "Use of Pozzolanic Materials in Mortars and Concretes" was held, consisting of eleven papers. The symposium is now in process of being published by the Society as a Special Technical Publication.

The committee records with sorrow the deaths of two of its long-time members, Joshua L. Miner and Thomas A. Hicks.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee C-1 presented to the Society, through the Administrative Committee on Standards, the following four tentatives:

Tentative Specifications for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 - 50 T),[2]

Tentative Method of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 - 50 T),[2]

Tentative Revision in Standard Method of Test for Heat of Hydration of Portland Cement (C 186 - 49),[3] and

Tentative Revision of Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 - 47).[2]

These recommendations were accepted by the Standards Committee on March 1, 1950, with the exception of the tentative revision of Standard Method of Test C 109, which was accepted on September 22, 1949.

ADDITIONS ACCEPTABLE UNDER SPECIFICATIONS C 175 T

Based on the recommendations of the Working Committee on Additions, Committee C-1 has declared the following two materials acceptable for inclusion in the list of air-entraining agents acceptable under Specifications C 175 T:

1. A material known commercially as N-TAIR, which is manufactured by Newport Industries, Inc., and consisting substantially of a sodium resinate produced from pine wood stumps from which the bulk of the petroleum naphtha-soluble resin acids has been removed. The resin, if regenerated from the soap, has an acid number not less than 125. The N-TAIR has been stabilized so that it will remain homogeneous and completely soluble at all working concentrations.
2. A material known commercially as Aira-

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1950 Supplement to Book of ASTM Standards, Part 3.

[3] 1949 Book of A.S.T.M. Standards, Part 3.

lon, which is manufactured by the Dewey & Almy Chemical Co., and which consists substantially of hydro aromatic and fatty carboxylic acids, being derived from an alkaline process of paper manufacture and neutralized to make a water-soluble soap. The acids, if regenerated from the soap, have an acid number of 150 to 160.

ACTIVITIES OF SUBCOMMITTEES

The Sponsoring Committee on Portland Cement (R. R. Litehiser, chairman) has been engaged in studies of questions relating to the specifications under its jurisdiction. Particular attention was paid to the relative merits of the tensile and compressive strength tests, and the subcommittee recommended that a footnote under Table II of Specifications C 150 for Portland Cement be revised to the effect that, in the absence of preference being indicated by the purchaser, the compressive strength requirement would govern. However, the recommendation was not favorably acted upon by Committee C-1.

The Sponsoring Committee on Masonry Cement (H. D. Baylor, chairman) is making further study of the specifications for natural cement and masonry cement. Efforts are being made to improve the methods of testing the natural cement for strength and autoclave expansion.

The Sponsoring Committee on Blended Cement (L. R. Forbrich, chairman) is engaged in a series of cooperative tests in the search for a suitable means of measuring the pozzolanic activity of materials. A current study is directed towards the achievement of tentative specifications for certain pozzolanic materials.

The Working Committee on Methods of Chemical Analysis (W. C. Hanna, chairman) has contributed additional data on the results reported by three laboratories on nine samples of portland cement when using the flame photometer method for determining the alkali content. The subcommittee is also engaged in the study of an improved method for removing manganese in the precipitation of calcium.

The Working Committee on Volume Change and Soundness of Portland Cement (H. F. Gonnerman, chairman) presented a method of test for determining the potential alkali reaction of cement-aggregate combinations. That method has been recently submitted to the Society by Committee C-1 and was accepted by the Society as a new tentative. The subcommittee is engaged in assembling and studying data relating to the present specification limit on autoclave expansion of cement and is also preparing revisions of the bibliography relating to the subcommittee's work. Details of test equipment are being studied.

The Working Committee on Sulfate Resistance (D. Wolochow, chairman) has been engaged in a series of cooperative tests planned to provide the basis for a sulfate resistance performance test. Seventeen laboratories have been testing 13 cements, with and without air-entraining agents. Preliminary data are being studied. Four laboratories plan to make the sulfate susceptibility test as detailed in R. H. Bogue's book, "The Chemistry of Portland Cement."[4]

The Working Committee on Time of Set (E. E. Berger, chairman) developed a program of study of proposed test methods and has been engaged in a second cooperative series of tests on neat cement pastes.

The Working Committee on Heat of Hydration (W. J. McCoy, chairman) has been studying a new simplified heat of solution method for determining the heat of hydration of portland cement, and has planned a cooperative series of tests thereon. The new method was proposed by Leo Shartsis of the National Bureau of Standards. The subcommittee presented to the committee a new section governing conditions of retest that was accepted by the Society as a tentative revision of Method C 186, as mentioned earlier in this report.

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[4] R. H. Bogue, "The Chemistry of Portland Cement."

The Working Committee on Bleeding, Plasticity and Workability (H. L. Kennedy, chairman) presented a proposed Method for Measuring the Bleeding of Cement Pastes and Mortars, the method being based on the liquid-displacement principle that was described in the paper presented at the 1949 Annual Meeting of the Society by R. E. Valore, Jr., J. E. Bowling, and R. L. Blaine,[5] of the National Bureau of Standards. The proposed method is now being recommended by Committee C-1 as a new tentative, and is appended to this report.[6]

The Working Committee on Fineness (R. L. Blaine, chairman) has been engaged in studying questions related to the various fineness tests, including the study of air-permeability tests at lower pressures, and the question of extending the Wagner turbidimeter tests down to particles of portland cement of 2.5-micron size.

The Working Committee on Strength (G. L. Lindsay, chairman) has been studying questions related to the strength tests of mortars. The subcommittee proposed certain revisions designed to effect improvement in details of procedure and equipment used in Method C 109. These revisions were later accepted by the Society as tentative revisions, as mentioned previously in this report, and were appended as information in the latest printing of the method. The subcommittee has just recently submitted to the committee additional proposed revisions in Method C 109, as described later in this report.

The Working Committee on Additions (C. H. Scholer, chairman) had reviewed the results of tests on two materials that had been submitted to Committee C-1 as additions to cement under Tentative Specifications C 175. Based on the subcommittee's recommendations, Committee C-1 declared that those two materials were acceptable for inclusion among the acceptable additions listed in Tentative C 175. These actions have been reported to the Society, and have been recorded in the ASTM BULLETIN. The two additions are described earlier in this report. The subcommittee also prepared and submitted a proposed specification for air-entraining additions used in making air-entraining portland cements. The proposed specification was later accepted by the Society as new Tentative Specifications C 226 T, mentioned in this report and appended thereto.[2] The new tentative marks an important development in the work of Committee C-1. When and if the use of this new tentative is incorporated as a revision of Specifications C 175 T and C 205 T, there will no longer exist the need for the committee to pass on the acceptability of individual air-entraining additions, as is now required by Specifications C 175 T, and additions would not be mentioned by their commercial names, as is now done under the notes to that specification.

The Working Committee on Coordination of Methods of Test (J. R. Dwyer, chairman) has continued its study of questions of common interest to some of the various subcommittees, particularly the questions relating to flow tables and standard sand.

The Working Committee on SO_3 Content (H. S. Meissner, chairman) submitted two lengthy progress reports on the results being obtained in its current extensive cooperative study of eight cements, each prepared with seven different amounts of gypsum. The second report summarized the results of the effect of SO_3 content on durability as revealed by freezing-and-thawing tests. Therein, the subcommittee pointed out that the optimum SO_3 content, as defined by strength and volume change of mortars, is slightly less than the SO_3 content for maximum durability. In the presence of air-entraining agents, however, the differences in durability due to varying the SO_3 content appeared to be negligible. The subcommittee is currently busy in a cooperative study of a convenient performance test designed to show when a cement has the optimum of gypsum.

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 891 (1949).

[6] The new tentative method was accepted by the Society and appears in the 1950 Supplement to ASTM Book of Standards, Part 3.

The Working Committee on Methods of Test for Air-Entrainment (H. K. Cook, chairman) continued its study of questions related to its work, and has outlined a very extensive series of cooperative tests for the near future.

The Subcommittee on Cement Reference Laboratory (G. E. Warren, chairman) has continued its assistance in guiding the work of the Cement Reference Laboratory at the National Bureau of Standards. This joint project of the Government and the A.S.T.M. is sponsored by Committee C-1, and is supported by funds received through the National Bureau of Standards, the Bureau of Public Roads, and the A.S.T.M. Progress has been made in rebuilding and training personnel. The laboratory inspections during the past year have included the verification of compression testing machines up to the capacity of 200,000 lb. Additional data have been secured in cooperative tests of the experimental mixture prepared by the Laboratory for the calibration of flow tables, and the question of making such a sample more generally available to interested laboratories is now being studied. The Laboratory has cooperated with the various A.S.T.M. subcommittees in matters relating to apparatus and methods that are of possible interest in testing cement.

The Editorial Committee (W. J. McCoy, chairman) has continued its study of the various standards that are under the jurisdiction of Committee C-1 and has presented recommendations to the appropriate subcommittees.

NEW TENTATIVE

Tentative Method of Test for Determining the Bleeding of Cement Pastes and Mortars.—Because of the long-existent interest in the bleeding of cements in the various mixtures in which they are used, the committee recommends the acceptance as tentative of this method, as appended to this report.[6] The application of this liquid-displacement method to the collection and continuous measurement of bleeding water formed the subject of a paper by Valore, Bowling and Blaine at the 1949 Annual Meeting of the A.S.T.M.[5] Cooperative study has recently been devoted to this subject by a number of laboratories. No recommendations are made as to limits, and limits on bleeding are not included in current A.S.T.M. cement standards. It is hoped that the publication as tentative will promote the study of a question of great interest to makers and users of cement.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 – 49).—The committee recommends revisions for immediate adoption in this standard, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society. It is advantageous to have these revisions become effective at the same time the recommended advancement of the existing tentative revision may be made, so that certain refinements in details of the test may be accomplished at one time. The revisions proposed for immediate action are as follows:

Section 2 (g).—Replace the entire section with the following sentence: "The flow table shall conform to the requirements of Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (A.S.T.M. Designation: C 230)."

Section 2 (h).—Replace the entire section with the sentence: "The flow mold shall conform with the requirements of Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cement (A.S.T.M. Designation: C 230)."

Section 2 (i).—Add the following sentence at the end of this section: "The tamping face of the tamper shall be flat and at right angles to the length of the tamper."

Section 8 (b).—Omit the word "stirring" from the first sentence of this section. In the second sentence change item (1) to read as follows: "(1) Place the water in the dry bowl."

Section 9.—Change the ninth sentence to read: "The flow is the resulting increase in average diameter of the mortar mass, measured on at least four diameters at approximately equi-spaced intervals, expressed as a percentage of the original diameter."

ADOPTION OF TENTATIVE REVISION AS STANDARD

Standard Method of Test for Compressive Strength of Hydraulic-Cement Mortars (C 109 - 49).—As already mentioned in this report, the Society accepted certain tentative revisions in C 109 during the past year. These revisions were designed to effect certain refinements in the apparatus and procedure used in that test. Those revisions were appended to Methods C 109[3] in the latest printing of the method. Apparently, the revisions were favorably regarded, and now the committee recommends the adoption as standard of the tentative revision, with the exception that the revision proposed for the ninth sentence of Section 9 is to be disregarded, that sentence now being covered by the new revision, elsewhere recommended in this report for immediate adoption as standard.

TENTATIVES CONTINUED WITHOUT REVISION

Because of related current study, the committee recommends that the following tentatives be retained as tentative without revision at this time:

Tentative Methods of Chemical Analysis of Portland Cement (C 114 - 48 T)[3]
Tentative Specifications for Air-Entraining Portland Cement (C 175 - 48 T)[3]
Tentative Method of Test for Fineness of Portland Cement by Air-Permeability Apparatus (C 204 - 46 T)[3]
Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 - 48 T)[3]
Tentative Definition of Term Addition as Applied to Hydraulic Cements (C 219 - 48 T)[3]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.

The election of officers for the ensuing term of two years resulted in the election of the following:

Chairman, R. R. Litehiser.
Vice-Chairman, W. C. Hanna.
Secretary, W. S. Weaver.
Six Members at Large of the Advisory Committee, H. D. Baylor, R. F. Blanks, F. H. Jackson, W. H. Klein, G. L. Lindsay, C. H. Scholer.

This report has been submitted to letter ballot of the committee, which consists of 86 voting members, of whom 72 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. H. JACKSON,
Chairman.

G. E. WARREN,
Secretary,

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Air-Entraining Portland Cement (C 175 - 48 T)

Revision of Tentative Method of:

Test for Air Content of Air-Entraining Hydraulic-Cement Mortar (C 185 - 49 T).

Tentative Revision of Standard Specifications for:

Portland Cement (C 150 - 49).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the revised tentatives together with the tentative revision of standard C 150–49 appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-2
ON
MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS[1]

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements has met three times during the year.

Owing to the increasing interest in and the extended applications of oxysulfate cements, the scope of the committee has been broadened to include these products.

The following officers have been reelected for the ensuing term of two years:

Chairman, L. S. Wells.
Vice-Chairman, Dean Hubbell.
Secretary, G. J. Fink.

Seventeen methods of test were submitted to the Society with the recommendation that they be published as tentative. Three of these methods have been approved and appear in the 1949 Book of A.S.T.M. Standards. Revisions of the remainder, as suggested by the Administrative Committee on Standards, are under review. Additional methods are being considered by Subcommittee II on methods of test.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Test for Sieve Analysis of Plastic Calcined Magnesia (C 239 – 49 T),
Test for Sieve Analysis of Magnesium Oxychloride Compositions, Aggregates and Fillers (C 238 – 49 T), and
Sampling Magnesium Oxychloride Compositions and Ingredients (C 237 – 49 T).

These recommendations were accepted by the Standards Committee on January 17, 1950, and the new tentative methods appear in the 1949 Book of A.S.T.M. Standards, Part 3.

This report has been submitted to letter ballot of the committee, which consists of 17 voting members; 13 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

G. J. FINK,
Secretary.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

EDITORIAL NOTE

Subsequent to the 1950 Annual Meeting, Committee C-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

- Chemical Analysis of Magnesium Sulfate (Epsom Salts) Technical Grade, $MgSO_4 \cdot 7H_2O$ (C 244 – 50 T),
- Analysis of Magnesium Chloride ($MgCl_2 \cdot 6H_2O$) (C 245 – 50 T),
- Testing Magnesia (MgO) for Magnesium Oxychloride Cements (C 246 – 50 T),
- Determination of Ignition Loss and Active Calcium Oxide in Magnesium Oxide for Use in Magnesium Oxychloride Cements (C 247 – 50 T),
- Test for Bulk Density of Magnesium Oxychloride Cement Compositions (C 248 – 50 T),
- Slump Test for Field Consistency of Magnesium Oxychloride Cements (C 249 – 50 T),
- Test for Field Determination of the Specific Gravity of Gauging Solutions of Magnesium Chloride and Magnesium Chloride-Magnesium Sulfate for Magnesium Oxychloride Cement Compositions (C 250 – 50 T),
- Mixing Oxychloride Cement Compositions with Gauging Solution for Preparation of Specimens for Laboratory Tests (251 50 T),
- Determination of Linear Contraction of Manesium Oxychloride Cements (C 252 – 50 T),
- Determination of Linear Change of Magnemsium Oxychloride Cements (C 253 – 50 T),
- Determinaton of Setting Time of Magnesium Oxychloride Cements (C 254 – 50 T),
- Determinaion of the Consistency of Magnesium Oxychloride Cements by Means of a Flow Table (C 255 – 50 T)
- Test for Transverse Strength of Magnesium Oxychloride Cement Compositions Using Simple Bar with Knife Edge or Two Poading (C 256 – 50 T),
- Test for Compressive Strength of Magnesium Oxychloride Cement Compositions (C 257 – 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new tentative methods appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-4
ON
CLAY PIPE[1]

Committee C-4 held one meeting during the year on June 28, 1949, during the annual meeting of the Society.

The record of the year's events must chronicle with sadness the news of the deaths of Anson Marston and H. E. Kilgus. Both were members of Committee C-4 for many years and their generous contributions of time and effort to advance committee activities were continually helpful throughout the years. They will be missed from the meetings for their memory will remain indelibly impressed in the minds of all who knew them.

Dean Marston was for a long time chairman of the committee. A memorial resolution recording the committee's feeling of profound sympathy was prepared and was presented to Dean Marston's family.

Adoption of Tentative as Standard

The committee recommends that the *Tentative Specification for Standard Strength Perforated Clay Pipe* (**C 211 - 47 T**)[2] be approved without change for reference to letter ballot of the Society for adoption as standard.

Activities of Subcommittees

Subcommittee II on Specifications (J. O. Everhart, chairman) has held one meeting during the year. Consideration has been given to the following revisions in tentatives:

Tentative Specifications for Standard Strength Clay Sewer Pipe (**C 13 - 44 T**). — Eliminate the column entitled "Maximum Inside Diameter of the Socket at $\frac{1}{2}$ in. above the Base" in Table II.

Tentative Specification for Extra Strength Pipe (**C 200 - 44 T**).—The subcommittee recommended that a general revision of the Extra Strength Clay Pipe Specification should be made and to this end is making a thorough study and investigation in order to report its findings to Committee C-4 at the next meeting.

Recommended Practice for Laying Sewer Pipe (**C 12 - 19**).—Since there have been no changes made in C 12 - 19 for many years, Subcommittee II is studying it for possible revision to bring it up to date.

Subcommittee II has been given the task of writing a new specification for standard strength ceramic glazed pipe and for ceramic glazed extra-strength clay pipe. A small working committee under the chairmanship of E. C. Clemens has completed a draft of the standard strength ceramic glazed specification.

Subcommittee III on Clay Flue Lining (Wendell Anderson, chairman).—Organization of a new subcommittee on clay flue lining has been completed with a total membership of eight. Since the scope of Committee C-4 did not provide for the writing of a specification for flue lining, the wording pertaining to the scope was amended to include flue lining. Because of the complexity involved in drafting standards for clay flue linings, the subcommittee has recommended, and its members are undertaking, an investigation of all service factors necessary for formulating such a standard. This research is likely to extend over a considerable period of time.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 3.

This report has been submitted to letter ballot of the committee which consists of 32 members; 25 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. C. RIEDEL,
Chairman.

R. G. SCOTT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Standard Strength Clay Sewer Pipe (C 13 - 44 T), and
Extra Strength Clay Pipe (C 200 - 44 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the revised tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

On January 17, 1951, the Administrative Committee on Standards accepted the recommendation of Committee C-4 that the Tentative Specifications for Ceramic-Glazed Pipe be published as tentative. The new tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 3, bearing the designation C 261 - 50 T.

REPORT OF COMMITTEE C-7
ON
LIME[1]

Committee C-7 on Lime held two meetings during the past year: the first at Atlantic City, N. J., during the Annual Meeting of the Society, and the second at Pittsburgh, Pa., on March 3, 1950. The committee now consists of 45 members, of whom 22 are classified as producers, 7 as consumers, and 16 as general interest members.

Only one change in existing standards and tentatives was proposed during the year. A tentative revision of the Specifications for Hydraulic Hydrated Lime (C 141 – 42) was accepted by the committee, which would add another class of hydraulic lime with no change in the present specifications.

Progress was made in revising Specifications for Quicklime and Hydrated Lime for Water Treatment (C 53 – 39), and two new specifications for lime for calcium carbide and for grease manufacture respectively, were prepared and sent to letter ballot of the committee. A letter ballot, as a matter of information, was taken on the desirability of writing specifications on "agricultural liming material" with a majority of the committee not considering it favorably.

Considerable committee work has been performed on round-robin testing for different methods of analyzing impurities in lime, such as iron, CO_2, fluorine, and trace elements.

The following officers were elected for the ensuing term of two years:

Chairman, Walter C. Voss.
Vice-Chairman, L. K. Herndon.
Vice-Chairman, E. E. Eakins.
Secretary, Robert S. Boynton.

The report has been submitted to letter ballot of the committee, which consists of 45 members; 37 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

Walter C. Voss,
Chairman.

Robert S. Boynton,
Secretary.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

Editorial Note

Subsequent to the Annual Meeting, Committee C-7 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:
Quicklime andCalcium Carbide Manufacture (C 258 – 50 T), and
Hydrated Lime for Grease Manufacture (C 259 – 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new tentative specifications appear in the 1950 Supplement to Book of A.S.T.M. Standards, Part 3.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES[1]

Committee C-8 on Refractories has held two meetings during the past year: the seventy-sixth in Bedford Springs, Pa., on September 22, 1949, and the seventy-seventh in Pittsburgh, Pa., on February 28, 1950.

The committee has lost one member by death, Ralph M. Bowman. His place as representative of Republic Steel Corp. has been filled by the election of John J. Hazel. B. L. Dorsey has been elected as successor to the late L. A. Smith as representative of Jones & Laughlin Steel Corp. C. A. Varall succeeds W. S. Elliott as representative of the Department of Public Works, New York City.

The president, vice-chairman, and secretary were reelected for the ensuing term of two years.

The limit of membership of the committee was increased from 38 to 40, to permit the election of two members to represent the newer special refractories field. The present membership is 38, of whom 17 are classified as producers, 13 as consumers, and 8 as general interest members. There are 15 consulting members.

REVISION OF TENTATIVE

Tentative Specifications for Castable Refractories for Boiler Furnaces and Incinerators (**C 213 - 47 T**).[2]—The committee recommends the following revision of these specifications:

Section 9(c).—Replace the period in the last sentence with a comma and add: "except that when using a hydraulic testing machine the load shall be applied at the rate of 500 lb. per min." The purpose of this change is to bring the time of loading-to-failure in the modulus of rupture of castables into line with that of Types F and G brick and air-set mortars.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications for Refractories for Malleable Iron Furnaces with Removable Bungs, and for Annealing Ovens (**C 63 - 47**).[2]—The committee recommends the following tentative revision of this standard:

Section 11 (i).—Replace the period in the last sentence with a comma and add: "except that when using a hydraulic testing machine for modulus of rupture for Type I brick and shapes the load shall be applied at the rate of 1000 lb. per min."

Standard Methods of Chemical Analysis of Refractory Materials (**C 18 - 45**).[2]—The committee recommends the following tentative revisions of this standard:

Section 12.—Delete this paragraph and substitute the following:

> 12. Determine TiO_2 colorimetrically by use of a photometer as follows: Transfer the 25-ml. portion reserved for the determination of TiO_2 (Section 11) to a 100-ml. volumetric flask. Add 5 ml. of H_2SO_4 (1:1). Cool to room temperature and dilute to the mark. Transfer exactly half of the solution to another 100-ml. volumetric flask. Dilute one of the flasks to the mark with H_2SO_4 (1:19). To the other add 5 ml. of 3 per cent H_2O_2, then dilute nearly to the mark with H_2SO_4 (1:19), adjust temperature to 25 ± 1 C., then adjust volume exactly and let stand at least 5

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] **1949 Book of A.S.T.M. Standards, Part 3.**

min. Transfer a portion from the first flask to a cuvette and set the potentiometer scale reading at zero. Then measure the absorption of the solution in the second flask. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solutions to H_2SO_4 (1:19), develop color with 5 ml. of 3 per cent H_2O_2, let stand 5 min. and read the absorption, using H_2SO_4 (1:19) for the zero setting of the potentiometer scale.

NOTE 1.—If a spectrophotometer is used, measure the absorption at mean transmission of 420 mμ. If a filter photometer is used, use a glass with a maximum transmission in the region of 420 mμ.

NOTE 2.—As an alternate method, TiO_2 can be determined in the 25-ml. portion reserved for this purpose (Section 11) by oxidizing both the sample and the standard TiO_2 solution with several drops of a H_2O_2 solution (30 per cent). Compare the colors either in Nessler tubes or in a suitable colorimeter. Use a H_2SO_4 solution (5 per cent) for diluting purposes in matching the colors.

Section 23.—Delete this paragraph and substitute the following:

23. Fuse the ignited R_2O_3 residue after recovery of SiO_2 (Section 21) with a small piece of $K_2S_2O_7$ or $Na_2S_2O_7$. Cool, and dissolve the melt in 30 ml. of H_2SO_4 (1:5). Transfer to a 100-ml. volumetric flask, and dilute almost to the mark. Cool to 25 ± 1 C. and adjust volume exactly. Transfer a portion of the solution to a photometer cuvette and set the potentiometer scale reading at zero. To the balance of the solution in the flask add 5 drops of 30 per cent H_2O_2, mix, let stand at least 5 min., and measure the absorption of the colored solution. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solution to H_2SO_4 (1:19), develop color with 30 per cent H_2O_2, let stand 5 min., and read the absorption, using H_2SO_4 (1:19) for the zero setting of the potentiometer scale.

NOTE 1.—If a spectrophotometer is used, measure the absorption at mean transmission of 420 mμ. If a filter photometer is used, use a glass with maximum transmission in the region of 420 mμ.

NOTE 2.—As an alternate method fuse the ignited R_2O_3 residue after recovery of SiO_2 (Section 21) with a small piece of $K_2S_2O_7$ or $Na_2S_2O_7$. Cool, and dissolve the fusion by the addition of about 25 ml. of warm water and 1 to 2 ml. of H_2SO_4 to the crucible. It is important at this point to keep the solution to a small volume. Transfer to a 50-ml. volumetric flask, add a few drops of H_2O_2, and dilute to the mark. Place the 50 ml. of solution in a small Nessler tube. Compare the color of this solution with the color of a known standard solution. A satisfactory solution is one of such strength that 1 ml. equals 0.0001 g. of TiO_2. To make the comparison use any standard colorimeter, or place an appropriate amount of standard solution in a second Nessler tube and dilute with water from a buret until the color is matched. From the amount of water added, calculate the percentage of TiO_2 in the sample.

Section 33.—In line 21 change "5.0 ml." to read "10.0 ml. (or 5.0 ml. if the alternate method is followed)".

Section 34.—Delete this paragraph and substitute the following:

34. Add to the 10.0-ml. portion previously reserved (Section 33) 10 ml. of H_2SO_4 (1:1) and evaporate to fumes. Dilute with 10 to 20 ml. of water, and cool. Transfer to a 100-ml. volumetric flask, and dilute to the mark. Transfer exactly half of the solution to another 100-ml. volumetric flask. Dilute one of the flasks to the mark with H_2SO_4 (1:19). To the other add 5 ml. of 3 per cent H_2O_2, then dilute nearly to the mark with H_2SO_4 (1:19), adjust temperature to 25 ± 1 C., then adjust volume exactly and let stand at least 5 min. Transfer a portion from the first flask to a photometer cuvette and set the potentiometer scale reading at zero. Then measure the absorption of the solution in the second flask. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solutions to H_2SO_4 (1:19), develop color with 5 ml. of 3 per cent H_2O_2, let stand 5 min. and read the absorption, using H_2SO_4 (1:19) for the zero setting of the potentiometer scale.

NOTE 1.—If spectrophotometer is used, measure the absorption at mean transmission of 420 mμ. If a filter photometer is used, use a glass with maximum transmission in the region of 420 mμ.

NOTE 2.—As an alternate method add to the 5.0-ml. portion previously reserved (Section 33) 10 ml. of H_2SO_4 (1:1) and evaporate to fumes. Dilute with 10 to 20 ml. of water, and cool. Pour into a 100-ml. volumetric flask, add a few drops of H_2O_2 (30 per cent), dilute to the mark, and mix. To a 100-ml. volumetric flask, add exactly 20.0 ml. of the standard TiO_2 solution and 10 ml. of H_2SO_4 (1:1). Add a few drops of H_2O_2 (30 per cent), dilute to the mark, and mix.

Compare the colors either in Nessler tubes or in a suitable colorimeter.

Section 55(b).—Change the last sentence to read as follows:

Finally dissolve the precipitate with hot H_2SO_4 (1:19) using a total of 125 ml. for dissolving and washing, and catching the solution and washings in a 250-ml. volumetric flask. Cool, and dilute to the mark. In case the alternate method is followed, dissolve the precipitate with hot diluted H_2SO_4 (10 per cent), cool, add several drops of H_2O_2, and dilute to 250 ml.

Section 56.—Delete this paragraph and substitute the following:

56. Transfer two 50-ml. aliquots to 100-ml. volumetric flasks. Dilute one flask to the mark with H_2SO_4 (1:19). To the other flask add 5 ml. of 3 per cent H_2O_2, and dilute nearly to the mark. Adjust the temperature to 25 ± 1 C. then make final volume adjustment. Let stand at least 5 min. before reading absorption. Transfer a portion from the first flask to a photometer cuvette and set the potentiometer scale reading at zero. Then measure the absorption of the solution in the second flask. Read the percentage of titania present from a calibration curve. Construct this curve by adding varying amounts of the standard titania solution to H_2SO_4 (1:19), develop color with 5 ml. of 3 per cent H_2O_2, let stand 5 min. and read the absorption, using H_2SO_4 (1:19) for the zero setting of the potentiometer scale.

NOTE 1.—If a spectrophotometer is used, measure the absorption at mean transmission of 420 $m\mu$. If a filter photometer is used, use a glass with maximum transmission in the region of 420 $m\mu$.

NOTE 2.—As an alternate method take 100-ml. aliquot portion from the 250-ml. acid solution (Section 55) and determine the TiO_2 colorimetrically by placing the solution in a 100-ml. Nessler tube and comparing it with the color of an appropriate amount of the standard TiO_2 solution in another Nessler tube. Both the solution of the sample and the reference standard solution shall be cooled to room temperature before making the comparison. Dilute the reference standard with a measured amount of water from a buret until the color is matched. From the amount of water added, calculate the percentage of TiO_2 in the sample. A standard colorimeter may be used instead of the Nessler tubes.

Standard Definitions of Terms Relating to Refractories (**C 71 - 47**).[2]—The committee recommends the following tentative additions to this standard:

Carbon Refractory.—A manufactured refractory comprised substantially or entirely of carbon (including graphite).

Carbon-ceramic Refractory.—A manufactured refractory comprised of carbon (including graphite) and one or more ceramic materials such as fireclay and silicon carbide.

The recommendations in this report have been submitted to letter ballot to the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman).—Surveys of current practice in the use of refractories in the copper, coke, and flat glass industries are expected to be ready for the next manual. Other surveys are in various stages of preparation.

Subcommittee II on Research (R. E. Birch, chairman).—A revised list of 25 research problems connected with tests on refractories is now available to anyone interested.

Subcommittee III on Tests (S. M. Phelps, chairman).—This subcommittee has approved the recommendations on which Committee C-8 based the actions on methods of test given earlier in this report.

Section A on Load (L. J. Trostel, chairman) recommends that with hydraulic machines a uniform rate of loading continue to be used. It favors single-point loading for the present, but recommends that the Research Subcommittee make a study of third-point loading. Reports from 22 laboratories showed hydraulic machines in use by 13 and mechanical machines by 12; further study is desirable on correlation of the two methods and on ways of specifying rates of loading.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Section B on Spalling (R. S. Bradley, chairman) was requested to look further into the question of spalling tests on specimens less than $2\frac{1}{2}$ in. thick; also mortars for bedding brick, in the standard test.

Section C on Temperature (J. L. Carruthers, chairman) reports that 23 companies, covering 29 plants and laboratories, are cooperating in the comparative P.C.E. test.

Section E on Chemical Analysis (H. A. Heiligman, chairman) is looking further into methods of determining loss on ignition of magnesite refractories containing lime.

Section F on Tests for Refractory Insulation (W. L. Stafford, chairman) has assembled data on rates of loading in modulus of rupture tests, but is withholding recommendations until a similar study on crushing strength shall have been completed.

Section I on Carbon Monoxide Disintegration (H. M. Kraner, chairman) has prepared a tentative method of test which is being published as information as an appendix to this report.

Subcommittee VII on Specifications (J. D. Sullivan, chairman) concludes from an inquiry, to which 9 consumers of raw fire clay replied, that a comprehensive specification covering other uses for fire clay than as mortar for laying up fireclay brick is not warranted at present.

Subcommittee XI on Special Refractories (H. Wilson, chairman) has outlined its field of activity as those refractories which are not as yet classified or defined, or for which no other subcommittee has been recognized within Committee C-8. It therefore covers dolomite, forsterite, spinel, cordierite, crystalline alumina, various carbides, so-called "mullite", diaspore and bauxite (unfused), zircon, and zirconia. Definitions and classifications are being assembled for submission to Subcommittees VI and IX.

Subcommittee XIV on Semi-Silica Brick (G. B. Remmey, chairman) has assembled samples for comparison by load test and P.C.E. test, with the expectation that the results will throw light on the best method of classifying fireclay brick containing a high percentage of silica.

This report has been submitted to letter ballot of the committee, which consists of 38 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. B. SOSMAN,
Chairman.

R. S. BRADLEY,
Vice-Chairman.

W. R. KERR,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-8 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Method for:

Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 – 49),
Panel Spalling Test for High Heat Duty Fireclay Brick (C 107 – 47),
Panel Spalling Test for Super Duty Fireclay Brick (C 122 – 47), and
Spalling Test for Fireclay Plastic Refractories (C 180 – 49).

These recommendations were accepted by the Standards Committee on January 19, 1951, and the tentative revisions appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

APPENDIX

PROPOSED METHOD OF TEST FOR DISINTEGRATION OF FIRECLAY REFRACTORIES IN AN ATMOSPHERE OF CARBON MONOXIDE[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test is intended to show the comparative behavior of fireclay refractories under the disintegrating action of carbon monoxide. The test is intended to accelerate service conditions for the purpose of determining in a relatively short time the probable behavior of the materials in service.

Apparatus

2. The apparatus and equipment shall consist of the following:

(*a*) *Heated Chamber.*—The chamber shall be gastight and of a suitable size, made of stainless steel, brass, aluminum, or unoxidized monel metal. A suggested size is 18 in. in diameter and 36 in. long. The unit may be heated by resistance wire or other means, provided that at the temperature of operation the difference in temperature between any two points within the chamber shall not be greater than 20 F. The chamber may be provided with a thermocouple well and shall have a gas inlet and outlet, with a provision for gas sampling at the gas outlet.

(*b*) *Temperature Control Instrument.*—The temperature of the test chamber shall be controlled and recorded by a suitable instrument having the required accuracy.

(*c*) *Atmosphere Control.*—The carbon monoxide shall be supplied from a tank of the gas, or manufactured by the conversion of carbon dioxide. The pressure from a tank supply shall be reduced by a regulator made for that purpose, and the flow of gas adjusted by means of a sensitive needle or regulating valve. A flow meter shall be used in the line as an aid for regulating the flow. When tank carbon monoxide is used, iron carbonyl is present in the gas and may cause clogging of the inlet tube, in which case the carbonyl may be removed before the gas enters the chamber. A soda-asbestos (Ascarite) tower in the inlet line will remove the carbonyl, but this should be preceded by a drying tower to prevent moisture from getting into the soda-asbestos.

(*d*) *Carbon Dioxide Absorbent.*—A sufficient quantity of burned lime or CaO (Note 1) shall be placed in the furnace or in the circulating system of the carbon monoxide to absorb the carbon dioxide as it is produced. A commercial CO_2 absorbent (Cardoxide) (Note 2) can be used outside the test chamber for the absorption of CO_2 from the circulating gas.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee C-8 on Refractories. Published as information, June, 1950.

NOTE 1.—Five pounds of CaO theoretically will absorb the carbon dioxide from a gas mixture containing 1 per cent CO_2 flowing at the rate of 0.5 cu. ft. per hr. for 7000 hr.

NOTE 2.—This granular material, probably $Ca(OH)_2$, is very efficient but does have the disadvantage that it gives off water vapor which must be taken out of the circulating gas with a $CaCl_2$ tower.

(*e*) *Furnace Pressure Control.*—The unit shall be equipped with a bubbling bottle or sensitive gage as a means to control the pressure of the exhaust gas. A positive pressure shall be maintained throughout the test.

(*f*) *Gas Analyzer.*—A conventional Orsat gas analyzer shall be used for periodic determinations of the carbon dioxide content of the exhaust gas.

Test Specimens

3. (*a*) The size of the specimen shall be 4½ by 4½ by 2½ (or 3) in., 3 by 3 by 4½ in., a standard 9-in. straight brick (either 2½ or 3 in. thick), or a 9-in. soap brick.

(*b*) If a specimen is cut from a shape, it shall be cut so as many original surfaces as possible remain intact.

(*c*) The weight, dimensions, color, and iron spots of each specimen shall be noted before testing.

Procedure

4. (*a*) The heating chamber containing the dried specimens shall be brought up to the operating temperature of between 925 and 940 F. without the carbon monoxide atmosphere. After the specimens have attained test temperature, the atmosphere of the chamber shall be changed so that it shall contain at least 95 per cent CO.

NOTE.—This may be done in one of the following ways: A vacuum pump may be used to evacuate the air from the chamber to a pressure of about 100 mm. of mercury. Carbon monoxide gas is then introduced and the evacuation process repeated three times. The test shall be timed from the last evacuation. A second method consists of flushing the air from the system with nitrogen, after which the nitrogen should be flushed from the system with a fast flow of carbon monoxide.

(*b*) After the flow of carbon monoxide has been started, it shall be regulated to a flow of not less than 2 cu. in. of carbon monoxide per hour per cubic inch of specimen volume. Orsat analyses should be taken at least twice a day and the carbon monoxide content of the exit gas shall be maintained above 95 per cent. If a recirculating system is used, the carbon monoxide flow may be greater.

(*c*) The specimens shall be inspected at the end of some prescribed period or periods. Before each inspection a fast flow of nitrogen shall be passed through the furnace to flush out the carbon monoxide. After the carbon monoxide is removed, a slow flow of nitrogen shall be maintained during cooling. If half the specimens in any one sample show complete disintegration at the end of any test period, the test for that sample may be considered complete.

Report

5. (*a*) The condition of each specimen shall be recorded at each inspection and any chipping, spalling, cracking, or disintegration noted. Dimensions, weights, and spotting shall be recorded also.

(*b*) The following classifications shall be used:

(*1*) Unaffected (if no particles are spalled off and no cracking).

(*2*) Slight disintegration (if specimen has slight cracking or particles less than ⅛ in. in diameter are spalled off).

(*3*) Partial disintegration (if specimen shows considerable cracking or particles larger than ⅛ in. in diameter are spalled off).

(*4*) Complete disintegration (if specimen is broken into more than four large pieces).

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES[1]

Committee C-9 on Concrete and Concrete Aggregates has been active throughout the year. Three meetings were held: on June 30, 1949, during the Annual Meeting of the Society in Atlantic City, N. J.; on October 13, 1949, in San Francisco, Calif., during the Pacific Coast meeting of the Society; and on March 1, 1950, in Pittsburgh, Pa., during the Spring Group Meetings of the Society. In spite of these evidences of activity, the committee has no recommendations to offer with respect to new standards or revisions in existing standards.

On December 19, 1949, the committee suffered the loss, through death, of Harry D. Jumper, a long-time and valued member. Mr. Jumper was first an individual member and later a representative of the Consolidated Rock Products Co. of Los Angeles, where he was Chief Engineer.

In accordance with provisions of the committee's regulations, H. S. Mattimore was elected an honorary member of Committee C-9. Mr. Mattimore has participated actively in the affairs of the committee since 1918.

Even though the committee has no recommendations to offer, it should be of interest to refer to some of the activities under way. Its work is carried out in three groups of subcommittees: Group I, covering Administration; Group II dealing with Research; and Group III in charge of the preparation of Specifications and Test Methods.

Subcommittee Activities

Group I, Administration.—Group I subcommittees are under the jurisdiction of Fred Hubbard, vice-chairman of the committee. The subcommittees cover Finance, Papers and Symposia, Sanford E. Thompson Award, Editorial and Definitions, and Membership. The Committee on Papers and Symposia, under the chairmanship of C. W. Allen, has been of assistance to the Administrative Committee on Papers and Publications in the formulation of program items dealing with concrete and concrete aggregates. The Subcommittee on Editorial and Definitions, under the chairmanship of L. W. Teller, has under way an exhaustive editorial review of existing A.S.T.M. standards. The Sanford E. Thompson Award Committee conducted a careful review of papers eligible for the award but decided to make no recommendations this year.

Group II on Research is under the chairmanship of A. T. Goldbeck. The objective of Group II subcommittees is to conduct and stimulate research pertaining to concrete and concrete aggregates. For the purpose of orderly procedure, this work is conducted under the jurisdiction of the following subcommittees:

Subcommittee II-a on Evaluation of Data with Horace A. Pratt as chairman deals with the application of statistical analysis to research data and is concerned, for illustration, with such problems as the needed frequency of testing to obtain truly indicative results.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

Subcommittee II-b on Chemical Reaction of Aggregates in Concrete (W. C. Hanna, chairman).—Subcommittee II-b is concerned with the fact that the aggregates as well as the cement sometimes undergo chemical reactions within the concrete, which may be beneficial in that they may increase the strength or may be detrimental in causing undue expansion and pattern cracking in concrete. The committee thus far has drawn from the experience of a large number of investigations in the laboratory and in the field; through discussions within the group it is hoped that light will be thrown on these phenomena which eventually may lead to a series of investigations to obtain special data where a lack of needed information exists.

Subcommittee II-c on Elasticity and Volume Changes of Concrete (R. E. Davis, chairman).—The development of tests for modulus of elasticity, Poisson's ratio, and similar values needed for design purposes is the work of this subcommittee. Air entrainment, the use of admixtures, the pre-stressing of steel, all would seem to make investigations in this field increasingly desirable since new elastic values are undoubtedly produced. Volume changes and plastic flow are also properly a part of the work of this committee.

Subcommittee II-d on Durability of Concrete (C. H. Scholer, chairman).—This subcommittee is dealing with highly important problems the solution of which may lead to greatly increased durability of concrete structures. Some of the questions which should be answered are:

1. What is the mechanism which causes the failure of portland-cement concrete under normal outdoor exposure to weathering action?

2. What characteristics of the cement and aggregate, physical and chemical, affect resistance to weathering?

3. What are the effects of construction practices, handling, placing, finishing and curing upon concrete durability?

4. What are the chemical and physical reactions which cause concrete failure with certain cement-aggregate combinations?

It is important that tests for all types of durability be developed.

Subcommittee II-e on Dynamic Testing of Concrete (Harold Sweet, chairman).—This subcommittee is continuously seeking to improve this very interesting and valuable method for measuring the modulus of elasticity of concrete by dynamic methods which may be used to trace the failure of concrete during durability tests without actually destroying the specimen, thus permitting the test to continue. Already adaptations of this general method are leading to the testing of structures in place, and its application to the determination of the extent of cracking and of concrete deterioration is rapidly being developed.

Subcommittee II-f on Aggregate Mineralogical Characteristics as Related to Concrete (D. O. Woolf, chairman).—This subcommittee is making rapid progress in its work of writing descriptions of rock in terms acceptable to engineers. The committee is preparing descriptions of rocks commonly used in construction with a view of informing the engineer of the composition and physical characteristics of each material. In many cases these descriptions will be sufficiently complete to enable the engineer to identify the rock without the use of petrographic methods.

Many problems have been discussed by the Group II committees during the past year, and it is hoped that work on these problems may be undertaken as time permits. The following are typical problems for research:

1. Particle shape and its effect on concrete.
2. Development of a wearing test for concrete.
3. Effect of air-entraining agents on concrete (this is intended to include something more than their air-entraining effects).
4. Study of effect of gradation of aggregates on concrete.
    - (*a*) effect of changes in gradation of say ± 0.2 in fineness modulus.
    - (*b*) effect of maximum density *versus* other gradations.
    - (*c*) how serious are fluctuations in gradation?
    - (*d*) study of tolerance material in separated sizes.
5. Study of characteristics of aggregates on the durability performance of concrete.
    - (*a*) thermal characteristics.
    - (*b*) percentage of saturation.
    - (*c*) surface characteristics (roughness, coatings, etc.)
    - (*d*) diffusivity.
6. Study of sampling.

Group III on Specifications and Test Methods.—Twelve subcommittees comprise Group III of Committee C-9 on Concrete and Concrete Aggregates. All of these subcommittees are concerned with specifications or methods of tests for which standards or tentatives have been developed and are in use. Collectively, they have jurisdiction over 37 standards and tentatives. Each engages in studies of all matters within its field of activity, recommends revisions in existing standards and proposes new standards within its field. They are under the general chairmanship of R. R. Litehiser.

The field of activity of each of the twelve subcommittees is in general indicated by its title. However, each subcommittee conducts its work within a rather broadly defined scope. A list of the subcommittees and something about their current activity follows:

Subcommittee III-a on Methods of Testing Concrete for Strength (A. G. Timms, chairman) is responsible for six standards. Its primary current activity is in keeping these standards up to date. Last year the Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 - 49) was revised.

Subcommittee III-b on Methods of Testing Volume Changes of Concrete (C. P. Siess, chairman) is presently engaged in extensive revision of the one standard under its jurisdiction, Standard Method of Test for Volume Change of Cement Mortar and Concrete (C 157 - 43).

Subcommittee III-c on Methods of Testing Fresh Concrete (I. L. Tyler, chairman) has a particularly active schedule. It is currently engaged in studying revision of all six of the standards for which it is responsible. Its work last year culminated in the Tentative Method of Test for Air Content of Freshly Mixed Concrete by the Pressure Method (C 231 - 49 T).

Subcommittee III-d on Specifications for Concrete Aggregates (H. F. Clemmer, chairman).—Its work last year resulted in the revision of the one standard over which it has jurisdiction, Standard Specifications for Concrete Aggregates, (C 33 - 49).

Subcommittee III-e on Methods of Testing Concrete Aggregates for Physical Characteristics (C. W. Allen, chairman) is responsible for 14 of Committee C-9's 37 standards. While keeping its standards up to date alone is a sizable job, Subcommittee III-e developed a new tentative last year, Tentative Method of Test for Soft Particles in Coarse Aggregate (C 235 - 49 T).

Subcommittee III-f on Methods of Testing and Specifications for Lightweight Aggregates for Concrete (P. M. Woodworth, chairman) has one standard, Standard Specifications for Lightweight Aggregates (C 130 - 42). Revision of this standard in light of developments in its field constitutes the current work of this subcommittee.

Subcommittee III-g on Methods of Testing and Specifications for Curing Compounds (C. E. Proudley, chairman) has jurisdiction over two standards. Its work last year resulted in the revision of the Tentative Specifications for Waterproof Paper for Curing Concrete, (C 171 - 49 T). It is presently engaged in studies bearing on the revision of the Tentative Method of Test for Water Retention Efficiency of Methods for Curing Concrete (C 156 - 44 T).

Subcommitee III-h on Methods of Testing and Specifications for Admixtures (Bryant Mather, chairman).—Its work last year resulted in a new tentative, Tentative Methods of Testing Air-Entraining Admixtures (C 233 - 49 T). The subcommittee is currently engaged in revising this new tentative and in the preparation of Tentative Specifications for Air-Entraining Admixtures for Concrete. It is also lending assistance to the Sponsoring Committee on Blended Cements of Committee C-1 in the development of a Specification for Fly-Ash for use in concrete.

Subcommittee III-i on Methods of Testing and Specifications for Ready-Mixed Concrete (M. N. Clair, chairman) has the job of keeping Standard Specifications for Ready-Mixed Concrete (C 94 - 48) up to date.

Subcommittee III-j on Methods of Testing Concrete for Bleeding (W. H. Price, chairman) developed the one tentative standard, for which it is responsible, last year, the Tentative Method of Test for Bleeding of Concrete, (C 232 - 49 T). It is continuing its study in its field of activity.

Subcommittee III-k on Methods of Testing Concrete for Bond (H. J. Gilkey, chairman) introduced a new tentative last year, the Tentative Method of Test for Comparing Concretes on the Basis of Bond Developed with Reinforcing Steel (C 234 - 49 T). Its current work is concerned in keeping the one tentative standard for which it is responsible up to date.

Subcommittee III-l on Miscellaneous Tests of Hardened Concrete (H. F. Kriege, chairman) has two widely differing standards under its jurisdiction. Its work last year resulted in the revision of the Standard Method of Measuring Length of Drilled Concrete Cores (C 174 - 49). It is presently engaged in a study of the probable effect of reactive aggregates upon the Standard Method of Test for Cement Content of Hardened Portland-Cement Concrete (C 85 - 42).

This report has been submitted to letter ballot of the committee, which consists of 94 voting members plus 6 honorary members having voting privileges; 93 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectively submitted on behalf of the committee,

K. B. WOODS,
Chairman.

STANTON WALKER,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-9 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Air-Entraining Admixtures for Concrete (C 260 - 50 T).

Revision of Tentative Methods of:

Testing Air-Entraining Admixtures for Concrete (C 233 - 49 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-11
ON
GYPSUM[1]

Committee C-11 on Gypsum met on May 11 and 12, 1950, at the National Bureau of Standards, Washington, D. C. The committee now consists of 18 members, of whom 9 are classified as producers, 3 as consumers and 6 as general interest members.

The election of officers for the ensuing term of two years resulted in the election of the following:

Chairman, L. S. Wells.

Vice-Chairman, H. F. Gardner.

Secretary, L. H. Yeager.

The subcommittee structure of the committee has been reviewed and a reorganization was approved consisting of the following subcommittees:

Subcommittee I on Plasters; and

Subcommittee II on Structural Products.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee C-11 recommends that the following revisions of standards be approved for reference to letter ballot of the Society for immediate adoption and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting:

Standard Definitions of Terms Relating to Gypsum (**C 11 - 48**):[2]

Gypsum Concrete.—Change to read as follows:

> *Gypsum Concrete.*—A combination of aggregate or aggregates with calcined gypsum as a binding medium, which after mixing with water sets into a conglomerate mass.
>
> *Gypsum Fiber Concrete.*—Gypsum concrete in which the aggregate consists of shavings, fiber, or chips of wood.

Gypsum Lath.—Change to read as follows:

> *Gypsum Lath.*—A sheet or slab having an incombustible core, essentially gypsum, surfaced with paper suitable to receive gypsum plaster.
>
> *Perforated Gypsum Lath.*—Delete since the present text is not a definition and is to be included in Standard Specifications for Gypsum Lath (C 37 - 50).

Gypsum Molding Plaster.—Change to read as follows:

> *Gypsum Molding Plaster.*—A material consisting essentially of calcined gypsum for use in making interior embellishments, cornices, as gauging plaster, etc.

Gypsum Pottery Plaster.—Delete since the Standard Specifications for Gypsum Pottery Plaster (C 60 - 40) has been discontinued.

Gypsum Partition Tile or Block.—Delete Notes 1 and 2.

Gypsum Sheathing Board.—Change to read as follows:

> *Gypsum Sheathing Board.*—A sheet or slab having an incombustible core, essentially gypsum, surfaced with water-repellent paper.

Gypsum Wallboard.—Change to read as follows:

> *Gypsum Wallboard.*—A sheet or slab having an incombustible core, essentially gypsum, surfaced with paper suitable to receive decoration.

Perforated Gypsum Lath.—Delete this reference to Perforated Gypsum Lath.

Standard Specifications for Gypsum (**C 22 - 41**):[2]

Section 1 (*b*).—Change $CaSO_4 \cdot 2H_2O$ content from "64.5" to "70.0" per cent.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 3.

Standard Methods of Testing Gypsum and Gypsum Products (**C 26 – 42**):[2]

Delete the following sections and renumber subsequent sections accordingly:

Section 23.—Dry Bulk Procedure.

Section 24.—Wet Bulk.

Section 27 (a) and (b).—Tensile Strength, Apparatus.

Section 28 (a) and (b).—Test Specimen.

Section 29.—Procedure.

Section 30.—Report.

Section 31.—Add the following sentence:

Dry test specimens shall be placed in a desiccator over anhydrous calcium chloride for 24 hr., removed, and tested immediately.

Section 33.—Delete the last sentence. Add the following two sentences:

Where desirable to determine the tensile strength of neat plaster or any gypsum plaster mix, the apparatus described in Standard Method of Test for Tensile Strength of Hydraulic-Cement Mortars, (A.S.T.M. Designation: C 190) shall be used. The method of handling test specimens shall be as described above under "Compressive Strength."

Section 41.—Change to read as follows:

41. Two specimens, each 12 in. by approximately 16 in. shall be cut from each board in the sample, one having the 16 in. dimension parallel to the fiber of the surfacing material and the other at right angles thereto. The specimens shall be weighed to within one gram and then stored at a temperature of 70 to 100 F. in an atmosphere having a relative humidity of 25 to 50 per cent. The specimens shall be weighed once a day until the weight has become constant to within 0.1 per cent.

Section 42.—Change to read as follows:

42. Each specimen shall be centrally supported on fixed parallel bearings 14 in. on centers and the load shall be applied on a similar bearing midway between the supports. All bearings and load surfaces shall be true, shall engage the full width of the test specimen and shall be rounded to a radius of $\frac{1}{8}$ in. The test load shall be applied at a uniform rate of 60 lb. per min. with a permissible variation of plus or minus 10 per cent in the rate.

Section 43.—Change to read as follows:

43. The average breaking load of the specimens cut from the board in each direction shall be computed, and the results of the flexural strength tests shall be reported for the load applied across the fiber and the load applied parallel to the fiber of the surfacing.

Standard Specifications for Gypsum Plasters (**C 28 – 40**):[2]

Section 1.—Change the term "Gypsum ready-sanded plaster" to "Gypsum ready-mixed plaster" and the term "Calcined gypsum for finishing coat" to "Gypsum gauging plaster for finish coat".

Section 2.—Change to read as follows:

The calcined gypsum plaster in these specifications shall have a purity of not less than 66.0 per cent by weight of $CaSO_4 \cdot \frac{1}{2}H_2O$.

Section 3.—Change the heading from "Gypsum Ready-Sanded Plaster" to "Gypsum Ready-Mixed Plaster." Change Section 3 to read as follows:

Gypsum ready-mixed plaster is calcined gypsum plaster, mixed at the mill with mineral aggregate and other ingredients to control working quality and setting time.

Section 4.—Change to read as follows:

4. Gypsum ready-mixed plaster shall contain not more than 3 cu. ft. of mineral aggregate per 100 lb. of calcined gypsum plaster to which may be added fiber and material to control working quality and setting time. However, when prepared for application to porous masonry bases it may contain not more than 4 cu. ft. of mineral aggregate per 100 lb. of calcined gypsum plaster.

Section 5.—Change to read as follows:

Gypsum ready-mixed plaster shall have a time of set of not less than $1\frac{1}{2}$ nor more than 8 hrs.

Section 6.—Change the heading from "Tensile Strength" to "Compressive Strength" and revise to read as follows:

6. Gypsum ready-mixed plaster when dry shall have a compressive strength of not less than 400 psi. However, this strength requirement does not pertain to plasters prepared for application to porous masonry bases.

Section 7.—Change to read as follows:

Gypsum neat plaster is calcined gypsum plaster mixed at the mill with other ingredients to control working quality and setting time. Neat plaster may be fibered or unfibered. The addition of aggregate is required on the job.

Section 8.—Change to read as follows:

Gypsum neat plaster shall contain not less than 66.0 per cent by weight of $CaSO_4\cdot\frac{1}{2}H_2O$.

Section 10.—Change title from "Tensile Strength" to "Compressive Strength" and revise to read as follows:

10. Gypsum neat plaster as tested with two parts of standard Ottawa sand shall have a compressive strength of not less than 750 psi.

Section 12.—Change to read as follows:

12. Gypsum wood fiber plaster shall contain not less than 66.0 per cent by weight of $CaSO_4\cdot\frac{1}{2}H_2O$ and not less than 1.0 per cent by weight of wood fiber made from a non-staining wood.

Section 13.—Change to read as follows:

13. Gypsum wood fibered plaster shall set in not less than $1\frac{1}{2}$ nor more than 16 hr.

Section 14.—Change title from "Tensile Strength" to "Compressive Strength" and revise to read as follows:

14. Gypsum wood-fibered plaster when dry shall have a compressive strength of not less that 1200 psi.

Section 15.—Change the main heading from "Calcined Gypsum For Finishing Coat" to "Gypsum Gauging Plaster For Finish Coat." Change Section 15 to read as follows:

15. Gypsum gauging plaster is prepared for mixing with lime putty for the finish coat. It may contain materials to control setting time and working quality.

Section 16.—Replace with the following and renumber subsequent sections accordingly:

16. *Composition*.—Gypsum gauging plaster for finish coat shall contain not less than 66.0 per cent $CaSO_4\cdot\frac{1}{2}H_2O$ by weight.

Section 17.—Change to read as follows:

17. *Fineness*.—Gypsum gauging plaster for finish coat shall all pass a No. 14 mesh (1410-micron) sieve and not less than 60 per cent shall pass a No. 100 (149-micron) sieve.

Section 18.—Change to read as follows:

18. *Time of Setting*.—Gypsum gauging plaster for the finish coat....

Section 19.—Change the title from "Tensile Strength" to "Compressive Strength" and revise to read as follows:

19. Gypsum gauging plaster for finish coat shall have a compressive strength of not less than 1200 psi.

Standard Specifications for Gypsum Wallboard (**C 36 – 42**):[2]

Section 2.—Change the first two lines of Section 2 to read: "Gypsum wallboard shall consist of an incombustible core, essentially gypsum, with...."

Section 4.—Change to read as follows:

4. At least 0.25 per cent of the number of gypsum wallboards in a shipment, but not less than one board, shall be so selected as to be representative of the shipment, and shall constitute a sample for purpose of tests. When a shipment consists of more than one car or carrier load, a sample shall be taken from each car or carrier.

Section 5 (*a*).—Change second sentence to read as follows:

5(*a*). Edges of $\frac{3}{8}$ in. and $\frac{1}{2}$ in. board may be plain or recessed to receive a joint reinforcing strip; if recessed the depression shall not be less than 0.020 in. nor more than 0.045 in.

Section 5 (*b*).—Delete "36 in."

Section 5 (*d*).—Change maximum weights to read as follows:

Thickness, in.	Weight Per 1000 Sq. ft., Lb. Min.	Max.
$\frac{1}{4}$	900	1300
$\frac{3}{8}$	1350	1800
$\frac{1}{2}$	1800	2500

Section 6.—Change Notes 1 and 2 to read as follows:

NOTE 1.—Gypsum wallboard in thicknesses of $\frac{3}{8}$ in. and $\frac{1}{2}$ in. is available with square edges, recessed edges, and bevelled edges.

NOTE 2.—Gypsum Wallboard is available

with:

(*a*) The back surface covered with aluminum foil or other heat reflecting type of insulation.

(*b*) Pre-decorated face surface.

Standard Specifications for Gypsum Lath (**C 37 - 42**):[2]

Section 2.—Change first two lines to read: "2. Gypsum lath shall consist of an incombustible core, essentially gypsum with . . . ."

Section 3.—Delete all references to $\frac{1}{4}$ in. and $\frac{5}{16}$ in. lath in table.

Section 4.—Change to read as follows:

4. At least 0.25 per cent of the number of gypsum lath in a shipment, but not less than ten lath, shall be so selected as to be representative of the shipment, and shall constitute a sample for purpose of tests. When a shipment consists of more than one car or carrier load, a sample shall be taken from each car or carrier.

Section 5(a).—Delete reference to $\frac{1}{4}$ in. and $\frac{5}{16}$ in. thickness.

Section 5(b).—Change to read as follows:

Width.—The nominal width of gypsum lath shall be 16 in., 16$\frac{1}{5}$ in., 24 in. or 32 in. with permissible variations of $\frac{3}{16}$ in. under and $\frac{1}{8}$ in. over specified width.

Section 5(c).—Add the following sentence: "Lengths up to 12 ft. are available."

Section 5(d).—Change tabulation to read as follows:

Thickness, in.	Weight Per 1000 sq. ft., lb.	
	Min.	Max.
$\frac{3}{8}$	1350	1800
$\frac{1}{2}$	1800	2500

New Section 5(e).—Add a new paragraph to read as follows:

(*e*) *Perforated Gypsum Lath.*—Perforated gypsum lath shall meet the requirements of plain gypsum lath except that it shall have perforations not less than $\frac{3}{4}$ in. in diameter with one perforation for not more than 16 sq. in. of lath.

Section 6.—In the Note at the end of the section change "one surface" to read "back surface".

Standard Specifications for Gypsum Molding Plaster (**C 59 - 40**):[2]

Section 1.—Change to read as follows:

These specifications cover gypsum molding plaster, a material consisting essentially of calcined gypsum, for use in making interior embellishments, cornices, as gauging plaster, etc.

Section 2.—Change to read as follows:

Gypsum molding plaster shall contain not less than 80 per cent $CaSO_4 \cdot \frac{1}{2}H_2O$.

Section 4.—Change title from "Tensile Strength" to "Compressive Strength" and revise to read as follows:

Gypsum molding plaster shall have a compressive strength of not less than 1800 psi.

Standard Specifications for Keene's Cement (**C 61 - 40**):[2]

Section 1.—Insert the following sentence between the second and third sentences in the note as follows "It is generally available as quick setting and standard setting Keene's Cement."

Section 2.—Change to read as follows:

2. Keene's Cement shall set in not less than 20 min. nor more than 6 hr.

Section 3.—Change title from "Tensile Strength" to "Compressive Strength" and revise to read as follows:

3. Keene's Cement shall have a compressive strength of not less than 2500 psi.

Section 8.—In line 9, change "tensile" to read "compressive". In line 11, change "Section 15" to read "Section 19".

Standard Specifications for Gypsum Sheathing Board (**C 79 - 42**):[2]

Change Section 2 to read as follows:

2. Gypsum sheathing board shall consist of an incombustible core, essentially gypsum, with or without fiber, but not exceeding 15 per cent of fiber by weight, and surfaced with water repellent paper firmly bonded to the core.

Change Section 4 to read as follows:

4. At least 0.25 per cent of the number of gypsum sheathing boards in a shipment, but not less than one board, shall be so selected as to be representative of the shipment, and shall constitute a sample for purposes of tests. When a shipment consists of more than one car or carrier load, a sample shall be taken from each car or carrier.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee which consists of 17 members; 17 members returned their ballots, of whom 17 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. S. WELLS,
Chairman.

L. H. YEAGER,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the vote is on record at ASTM Headquarters.

REPORT OF COMMITTEE C-14
ON
GLASS AND GLASS PRODUCTS[1]

Committee C-14 on Glass and Glass Products held two meetings during the past year: at Shawnee-on-the-Delaware, Pa., October 6, 1949, and at New York City, April 26, 1950.

The following officers were elected for the ensuing term of two years:

Chairman, Leonard Ghering.
Vice-Chairman, Lee Devol.
Secretary, F. V. Tooley.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee C-14 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Sampling Glass Containers (C 224 – 49 T), and
Test for Resistance of Glass Containers to Chemical Attack (C 225 – 49 T).

Revision of Tentative Definition of:

Terms Relating to Glass and Glass Products (C 162 – 49 T).

These recommendations were accepted by the Standards Committee on July 27, 1949, and the new and revised tentatives appear in the 1949 Book of A.S.T.M. Standards, Part 3.

On March 1, 1950, the Administrative Committee on Standards accepted the recommendation of Committee C-14 that the Methods of Sampling and Testing Structural Non-load-Bearing Cellular Glass Blocks be published as tentative. The new tentative methods will appear in the 1950 Supplement to Book of A.S.T.M. Standards, Part 3, bearing the designation C 240 – 50 T.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends revisions for immediate adoption of three standards, as appended hereto,[2] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that the recommendations may be referred to letter ballot of the Society:

Standard Method for Hydrostatic Pressure Test on Glass Containers (C 147 – 43),

Standard Method of Polariscopic Examination of Glass Containers (C 148 – 43), and

Standard Method of Thermal Shock Test on Glass Containers (C 149 – 43).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definition (H. H. Holscher, chairman).—A proposed revision of the term "safety glass" in the Glass Glossary was approved by Committee C-14 during the year. Following a letter ballot within Subcommittee VIII on Flat Glass by which the members expressed the opinion that the revised definition of the term "safety glass" should be made consistent with the latest revision of the Federal Specification DD-G-451, an editorial change in the proposed revision was accepted and will appear in the 1950 Supplement to the Book of A.S.T.M. Standards, Part 3.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The revised methods were approved by the Society and appear in the 1950 Supplement to the 1949 Book of ASTM Standards, Part 3.

Subcommittee II on Chemical Analysis (F. W. Glaze, chairman).—The committee has under consideration tentatively a new procedure for the routine determination of boric oxide in glass. A number of other suggestions for projects have been received as a result of a poll of the committee and these will be given careful consideration by the committee.

Subcommittee III on Chemical Properties (J. F. Green, chairman).—Recent projects arising in this committee have fallen more logically into the scope of Subcommittee VII on Glass Containers and have been therefore referred to this committee. Because of the importance of water purity in chemical durability testing, Subcommittee III has established representation of Committee D-19 on Industrial water.

Subcommittee IV on Physical and Mechanical Properties (J. R. Littleton, chairman) has under consideration the possibility of standard procedures for softening, strain, and annealing point measurements.

Subcommittee VI on Glass Construction Block and Tile (A. H. Baker, chairman).—Tentative Methods of Sampling and Testing Structural Non-Load-Bearing Cellular Glass Blocks (C 240 - 50 T) was approved by the Administrative Committee on Standards, March 1, 1950.

Subcommittee VII on Glass Containers (K. C. Lyon, chairman).—Revisions of three standards, namely, C 147, C 148, and C 149 were submitted to the main committee for letter ballot, and approved for immediate adoption.

The Administrative Committee on Standards has approved during the year the Tentative Method of Sampling Glass Containers (C 224 - 49 T).

Subcommittee VII A on Pharmaceutical Containers (F. C. Flint, chairman).—The Administrative Committee on Standards approved during the year Tentative Methods of Test for Resistance of Glass Containers to Chemical Attack (C 225 - 49 T). The Administrative Committee on Standards recommended the consolidation of the three original test methods into one A.S.T.M. tentative.

Subcommittee VIII on Flat Glass (D. E. Sharp, chairman).—(Refer to the Subcommittee I report for some of the action of Subcommittee VIII during the year.) J. D. Ryan, C-14 representative on ASA Sectional Committee Z-2, reported during the year on the status of that committee's work on a test method for determining the abrasion resistance of plastics and safety glass. Mr. Sharp urged consideration of Mr. Ryan's proposal that the method, as related to glass, developed in Z-26 be studied for possible adoption by Committee C-14. Committee C-14 voted to create a new section of Subcommittee VIII for the purpose of studying the Z-26 method. C. H. Hahner was appointed by Mr. Sharp to head this special committee for conducting the necessary study.

This report has been submitted to letter ballot of the committee, which consists of 50 voting members; 29 members returned their ballots, of whom 27 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. W. TILLOTSON,
Chairman.

F. V. TOOLEY,
Secretary.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS[1]

Committee C-15 on Manufactured Masonry Units held two meetings during the past year: one meeting on June 28, 1949, at Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and a joint meeting with Committees C-7 and C-12 on October 13 in San Francisco, Calif.

The subcommittees of Committee C-15 have been quite active during the past year and have reviewed all tentatives and standards under their jurisdiction. With the exception of the following recommended changes, the subcommittees have recommended, and Committee C-15 concurs, that the existing tentatives and standards be continued without change.

The election of officers for the ensuing term resulted in the selection of the following:

Chairman, J. W. Whittemore.
First Vice-Chairman, Theodore I. Coe.
Second Vice-Chairman P. M. Woodworth.
Secretary, M. H. Allen.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Structural Clay Facing Tile (C 212 - 48 T).

Tentative Revision of Standard Specifications for:

Structural Clay Floor Tile (C 57 - 39), and
Structural Clay Non-Load-Bearing Tile (C 56 - 41).

These recommendations were accepted by the Standards Committee on September 22, 1949, and the revised tentative and tentative revisions appear in the 1949 Book of A.S.T.M. Standards, Part 3.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Specifications for Facing Brick (C 216 - 48 T)[2] be approved for reference to letter ballot of the Society for adoption as standard.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions[3] of the following standards be adopted as standard:

Standard Specifications for:

Building Brick (Made from Clay or Shale) (C 62 - 49), pertaining to Sections 2, 3 (*a*), 3 (*b*), and 4 (*a*).
Sewer Brick (Made from Clay or Shale) (C 32 - 42), pertaining to Section 3 (*a*) and Table I.
Structural Clay Load-Bearing Wall Tile (C 34 - 49), pertaining to Section 6 (*b*) and Table I.
Structural Clay Non-Load-Bearing Tile (C 56 - 49), pertaining to Section 6 (*b*) and the table in Section 2.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 3.

[3] Published with the individual standards in the 1949 Book of A.S.T.M. Standards, Part 3.

Structural Clay Floor Tile (C 57 – 49), pertaining to Section 6 (*b*) and Table I.

Methods of:

Sampling and Testing Brick (C 67 – 44), pertaining to Sections 3, 4, 10 (*c*), 11 (*d*), and new Sections 36 to 42 inclusive.

Definition of Terms Relating to:

Structural Clay Tile (C 43 – 49), pertaining to the Note under Plaster Base Finish and to Footnote 3.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.

This report has been submitted to letter ballot of the committee, which consists of 59 members; 43 members have returned their ballots, of whom 42 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. W. Whittemore,
Chairman.

Harry C. Plummer,
Acting Secretary.

Editorial Note

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Structural Clay Facing Tile (C 212 – 49 T), and
Grazed Masonry Units (C 126 – 44 T).

Tentative Revision of Standard Specifications for:

Concrete Building Brick (C 55 – 37),
Structural Clay Floor Tile (C 57 – 49),
Hollow Load-Bearing Concrete Masonry Units (C 90 – 44),
Structural Clay Load-Bearing Wall Tile (C 34 – 49),
Structural Clay Non-Load-Bearing Tile (C 56 – 49),
Hollow Non-Load-Bearing Concrete Masonry Units (C 129 – 39), and
Solid Load-Bearing Concrete Masonry Units (C 145 – 40).

Tentative Revision of Standard Methods of:

Sampling and Testing Concrete Masonry Units (C 140 – 39).

Revision and Reversion to Tentative of Standard Specifications for:

Drain Tile (C 4 – 24).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives together with the tentative revisions of the standards appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-16
ON
THERMAL INSULATING MATERIALS[1]

Committee C-16 on Thermal Insulating Materials and all of its subcommittees held two meetings during the past year. The first of these was the three-day fall meeting held at the Williamsburg Inn and Lodge, Williamsburg, Va., on September 28, 29, 30, 1949. The second meeting was held at the William Penn Hotel in Pittsburgh, Pa., on February 27 and 28 in conjunction with the spring group meeting of the Society. This second meeting was held on two days, which is according to the committee's wishes of having a three-day fall meeting and a two-day spring meeting. However, the crowded schedule of a two-day meeting, which eliminates the committee's dinner and the accompanying fellowship and which greatly decreases the amount of time for the members to discuss mutual problems, has brought forth many requests to return the spring meeting to the original three-day schedule. The committee, after a full discussion, unanimously voted to return to the three-day spring meeting schedule.

The committee, as of April 15, is composed of 3 consulting members, 63 voting members and 17 non-voting members. The voting membership is composed of the following: 28 producers, 22 consumers and 13 general interest members. During the past year several changes in membership of the committee have taken place due to resignations, but the committee has added 6 voting members and lost only 1 non-voting member.

At the spring meeting, the election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, E. R. Queer.
1st Vice-Chairman, H. W. Greider.
2nd Vice-Chairman, W. L. Scott.
Secretary, R. P. Sturgis.
Assistant Secretary, M. E. DeReus.
Assistant Secretary for Membership, E. C. Shuman.
Executive Committee Member at Large, A. B. Fisher.

Revision of Standard, Immediate Adoption

Committee C-16 recommends for immediate adoption the revised Standard Methods of Test for Thickness and Density of Blanket or Batt Type Thermal Insulating Materials, appended hereto[2], and which is a revision of A.S.T.M. Designation C 167 - 44[3] and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Committee C-16 further requests that the following definition be included in Standard Definition of Terms Relating to Thermal Insulating Materials (C168 - 48):[3]

Blanket or batt Insulation.—A flexible or semi-flexible thermal insulation composed of felted or matted fibrous materials either organic or inorganic, which may contain binder or may be reinforced with confining media and suitably bound together.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.**

[2] **This revised method was accepted by the Society and appears in the 1950 Supplement to ASTM Book of Standards, Part 3.**

[3] **1949 Book of A.S.T.M. Standards, Part 3.**

ACTIVITIES OF SUBCOMMITTEES

There has not been any change from previous reports in the scope of the work being undertaken by the various subcommittees. Changes in chairmanship of some of the subcommittees have been made because of membership changes or because some of the subcommittee chairmen were assuming new responsibilities in the committee. Listed are the subcommittees with their present chairman:

Subcommittee:

S-I on Block and Pipe Insulation, C. R. Hutchcroft, chairman,
S-II on Structural Insulating Board, S. M. VanKirk, chairman,
S-III on Insulating Cement, J. E. Gaston, chairman,
S-IV on Blanket Insulation, R. L. Davis, chairman,
S-V on Loose Fill Insulation, P. L. Rogers, chairman,
T-VI on Thermal Conductivity, J. D. Babbitt, chairman,
T-VII on Special Thermal Properties, G. M. Rapp, chairman,
S-VII on Dimensional Standards, W. H. Zane, chairman,
T-IX on Vapor Barriers for Insulation, R. H. Heilman, chairman,
A-X on Significance of Test, R. B. Crepps, chairman,
A-XI, Editorial, C. B. Bradley, chairman.
A-XII on Membership, E. C. Shuman, chairman.

This year concludes the first full year's activities after the reorganization of Committee C-16. Some subcommittees have had their accomplishments retarded due to changes in subcommittee chairmen but several have test methods completed, which could have been included in this report if delays had not been encountered. The Executive Subcommittee through the vice-chairman is expediting the issuance of Methods of test and product specifications with emphasis being placed on methods of test. The Significance of Test statement, which for a while appeared to be a stumbling block to the subcommittees, has not developed into the hurdle which it was first thought to be. The Planning Subcommittee, through its round-table discussions of problems facing the subcommittees, has been able to clarify and advise ways and means of eradicating some of the most controversial points. Most of these arise from striving for near perfection in standards which not only apparently slows up subcommittee action but also masks the visual evidence of the efforts being expended. This year should see many accomplishments in test methods and the results of the reorganization should become more apparent.

The effect of moisture on thermal conductivity has received increased interest recently, and a program is now being developed to enable testing to start as soon as possible. It is hoped that funds may be secured to finance this program so that testing equipment may be placed in operation before January 1, 1951.

This report has been submitted to letter ballot of the committee, which consists of 63 members; 60 members returned their ballots, of whom 48 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

RAY THOMAS,
Chairman.

W. L. SCOTT,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-17
ON
ASBESTOS-CEMENT PRODUCTS[1]

Meetings of Committee C-17 and its subcommittees were held in connection with the 1950 Annual Meeting on June 29, 1950. At the meeting, David Wolochow, National Research Council of Canada and a member of Committee C-17, presented a most interesting talk on "The Production, Properties and Uses of Asbestos Fiber."

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee C-17 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Asbestos-Cement Flat Sheets (C 220 - 49 T), and

Asbestos-Cement Corrugated Sheets (C 221 - 49 T).

These recommendations were accepted by the Standards Committee on July 27, 1949.

On September 22, 1949, the following recommendations were accepted by the Standards Committee:

New Tentative Specifications for:

Asbestos-Cement Roofing Shingles (C 222 - 49 T), and

Asbestos-Cement Siding Shingles and Clapboards (C 223 - 49 T).

The four new tentatives specifications appear in the 1949 Book of A.S.T.M. Standards, Part 3. Sections defining the significance of tests described in the specifications have been approved, subject to letter ballot of the committee, for submission to the Society as revisions of the above tentative specifications.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Methods of Test (C. R. Hutchcroft, Chairman).—Work on test methods for asbestos-cement pressure pipe is almost complete.

Subcommittee II on Specifications (M. V. Engelbach, Chairman).—This subcommittee is continuing its work of preparing specifications for asbestos-cement pressure pipe.

This report has been submitted to letter ballot of the committee, which consists of 32 voting members; 15 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

HUBERT R. SNOKE,
Chairman.

C. C. KELSEY,
Secretary.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

REPORT OF COMMITTEE C-18
ON
NATURAL BUILDING STONES[1]

Committee C-18 on Natural Building Stones is reporting for a two-year period. The committee held meetings in Washington, D. C., on April 18, 1949, and on March 17, 1950, both of which were preceded by meetings of the subcommittees. In addition Subcomittee I, Advisory, met on October 27, 1948, and on February 1, 1950, also in Washington, D. C.

The committee records with sorrow the death on June 14, 1949, of Ernest H. Nichols, Chief Engineer, The Funkhouser Co., a member of the Society for sixteen years and a member of the Subcommittees on Test Procedures and Specifications.

The membership of Committee C-18 now consists of 21 members, of whom 7 are classified as producers, 7 as consumers, and 7 as general interest members. There are also 2 consulting members.

An election of officers resulted in the re-election of the following:

Chairman, L. W. Currier.
Vice-Chairman, T. I. Coe.
Secretary, F. S. Eaton.

F. S. Eaton was appointed as representative of the Society on the ASA Sectional Committee A91 on Specifications for Building Granite, to succeed D. W. Kessler, who has been named representative of the National Bureau of Standards on the same sectional committee. F. S. Eaton was also named by Chairman Currier as representative of Committee C-18 on the Society's Committee E-7 on Non-Destructive Testing.

All standards under the jurisdiction of the committee have been reviewed and the committee presents recommendations as noted in the following paragraphs.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee C-18 presented to the Society through the Administrative Committee on Standards, the proposed Tentative Method of Test for Abrasive Resistance of Stone Subjected to Foot Traffic (C 241 – 50 T). This recommendation was accepted by the Standards Committee on May 8, 1950, and the new tentative is appended to this report.[2]

This method has had a long period of trial at the National Bureau of Standards and elsewhere, for determining the wear resistance of walking surfaces of stone or other hard materials. This test is currently used by many government agencies in specifications. This test has already proven of value in avoiding such conditions of uneven wear as are evidenced in the floor of the Union Station in Washington, D. C., where the red tiles are shown (by this test) to be three times as hard as the white.

ADOPTION OF TENTATIVE AS STANDARD

The following tentative is recommended for advancement to standard, with certain changes which are considered editorial in nature:

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The new method of test appears in the 1950 Supplement to ASTM Book of Standards, Part 3.

Tentative Definitions of Terms Relating to Natural Building Stone (C 119 – 48 T):[3]

Granite.—In the third sentence, change to read: "... spar molecule (Note 2), but 80," deleting the words "at least". In Note 2, delete the entire third sentence. Also delete "However," in the fourth sentence and designate this sentence as Note 3.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption this revision and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that it may be referred to the Society for letter ballot:

Standard Method of Test for Compressive Strength of Natural Building Stone (**C 170 – 46**):[3] The test method requires consideration from the standpoint of liberalization of the shape of the specimens. It is sometimes impossible to prepare specimens of desired shape from samples available. The following changes are recommended:

Section 4 (a).—In line 8 of Paragraph 4 (*a*), change word "shall" to "should."

Section 7 (b).—Change the introduction to this paragraph to read as follows: "(*b*) When the ratio of height to diameter (or lateral dimension) differs from unity by 25 per cent or more, the result shall be calculated to that of the corresponding cube, as follows:"

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following two standard methods of test because they are not serving a useful purpose today, such determinations now being seldom made and not appearing in present-day specifications. The committee wishes it clearly understood that the methods are not being withdrawn due to any defect in procedure.

Standard Method of Test for Modulus of Elasticity of Natural Building Stone (**C 100 – 36**),[3] and

Standard Method of Shear Testing of Natural Building Stone (**C 102 – 36**).[3]

STANDARDS CONTINUED WITHOUT REVISION

Committee C-18 recommends that the following standards be continued as standard, without revision:

Standard Method of:

Tests for Absorption and Bulk Specific Gravity of Natural Building Stone (**C 97 – 47**),
Flexure Testing of Slate (Modulus of Rupture, Modulus of Elasticity) (**C 120 – 48**),
Test for Water Absorption of Slate (C 121 – 48), and
Test for Modulus of Rupture of Natural Building Stone (C 99 – 47).

Method C 99 – 47 requires amendment to permit using other sizes of test specimens than that at present specified. It was proposed to modify Section 4 covering test specimens to lend written authority to what has long been laboratory practice. In the meantime, the committee reaffirms this test method in its present status, without change.

TENTATIVES CONTINUED WITHOUT REVISION

Committee C-18 recommends that the following tentatives be continued in their present status without revision:

Tentative Method of:

Test for Durability of Slate for Roofing (C 217 – 48 T), and
Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stone (C 218 – 48 T).

The recommendations appearing in this report have been approved by letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

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[3] **1949 Book of A.S.T.M. Standards, Part 3.**

[4] **The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.**

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (L. W. Currier, chairman) has held three meetings since the last published report. Continued progress and results are reported on its project of compiling nomenclature and definitions for the natural building stones. The subcommittee is currently engaged, with the granite producers, in formulating standard size range definitions of "texture" or "grain size" based on average sizes of the quartz and feldspar crystals making up the stone. Those definitions already compiled and in effect, C 119 - 48 and C 119 - 48 T, have proven their value both to the industry and to the U. S. Geological Survey.

At the meeting of March 17, 1950, it was voted to submit the present tentative definitions C 119 - 48 T to letter ballot of the full committee for recommendation for adoption as standard.

Subcommittee III on Test Procedures (D. W. Kessler, chairman) has held two meetings since the last report of Committee C-18. A test procedure for determining the abrasive resistance of stone subjected to foot traffic, has been accepted by the Society as a tentative method of test, as referred to earlier in this report. The means of reducing compressive strength results, when the test is made on a tall specimen, came under consideration. It was decided to change the wording of Section 7 (*b*) of the Standard Method of Test for Compressive Strength of Natural Building Stone (C 170 - 46) so that the results obtained on short specimens may be reduced to that of a cubical specimen also. The reduction formula works in both directions, and it is desirable to reduce all determinations to the same basis.

The test method for modulus of rupture or flexure was considered, with regard to an amendment making possible the employment of smaller test specimens than are at present specified. It was proposed to modify Section 4, Standard Method of Test for Modulus of Rupture of Natural Building Stone (C 99 - 47), to lend written authority to what has long been common practice. It was voted to study and check tests on record and present this proposal in 1951.

Subcommittee IV on Specifications (F. S. Eaton, chairman). Subcommittee IV met in April, 1949, and in March, 1950. A specification for exterior marble was submitted which is receiving further study. The granite industry group also has requested time for study and accumulation of necessary data on which to base and propose a specification for building granite.

Subcommittee V on Research (J. R. Shank, chairman).—Discussion has been held on the subjects of durability tests, the exposure test wall being sponsored jointly by the committee and the National Bureau of Standards as discussed in the Appendix to this report, problems presented by the granite facing of the Sather Tower in California, definitions and standards for surfaces and polish (tabled until further progress results from cooperative work with ASA Sectional Committee A 91), and a project for collecting study samples of weathered building stones to serve as checks in durability tests.

This report has been submitted to letter ballot of the committee, which consists of 21 voting members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. W. CURRIER,
Chairman.

F. S. EATON,
Secretary.

APPENDIX

EXPOSURE TEST WALL FOR NATURAL BUILDING STONES

A research project in the form of an exposure test wall has been undertaken cooperatively by the committee and the National Bureau of Standards. The wall was erected in a test plot on the property of the National Bureau of Standards during the latter part of 1948. The wall contains 2069 samples of stone in the front face and 231 more on the back and ends. Most of the stones are from domestic sources, and 47 states are represented. Samples from sixteen foreign countries are included.

The wall is mainly for the purpose of studying various phases of weathering on a large variety of stones under known conditions of exposure. Certain variables were introduced for the purpose of gain-

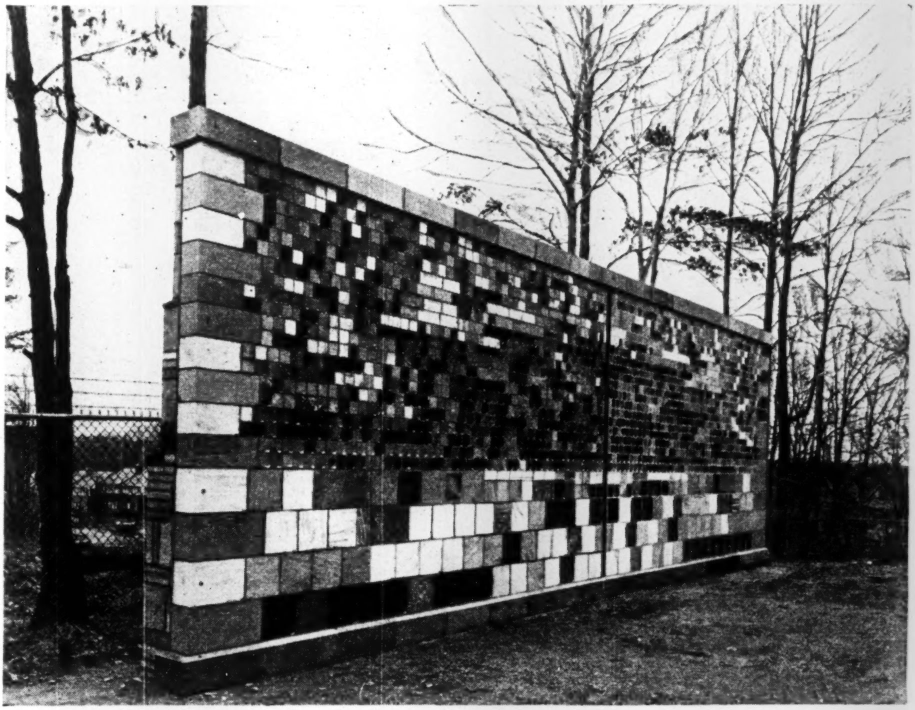

FIG. 1.—Exposure Test Wall.

The exposure test wall was erected at the National Bureau of Standards in 1948 for the purpose of studying phases of weathering on a large variety of building stones under known conditions of exposure.

ing information on certain construction methods. Stonework on the front of the wall was set with two kinds of mortar for studies on discoloration effects, the right-hand half being set in lime mortar and the other half with portland-cement mortar. Grade weatherproofings were placed under three sections of the granite base while two intervening sections have none. The back of the stonework in the central portion was back waterproofed before the concrete backing was poured, while the two remaining portions were not so treated. Certain arrangements of types of stone were made to test a theory that some combinations of stone cause more rapid weathering. Two large areas have dolomitic stones above calcareous stones and two other areas have calcareous stones above siliceous stones.

On the back of the wall are several panels and samples of thin veneer stone set in portland-cement concrete. Several of the thin stones were not cleaned of saw rust on the back faces before setting in order to determine if such practice contributes to discolorations. There are two watertables on the back, one covered with flagstones and the other with marble. Under each vertical joint in the marble watertable a thin piece of slate was placed in order to determine if such practice will prevent discolorations below the joints.

A vault is provided in the back of the wall for storing records and samples taken from various blocks before they were set. It is planned to keep the test under observation for many years. When blocks show definite amounts of deterioration they will be removed from the wall and replaced by new samples. By laboratory studies on the weathered stones, efforts will be made to identify the causes and extent of weathering. The project is mainly an effort to determine the various causes of deterioration in stone masonry and to improve our durability tests.

REPORT OF COMMITTEE C-19
ON
STRUCTURAL SANDWICH CONSTRUCTION[1]

Committee C-19 has held two meetings during the year: on September 30, 1949, and April 4, 1950, both in Philadelphia, Pa. Since the last report seven new members have been elected and two members have resigned. The total membership of the committee is now 57, of whom 47 are voting members; 20 are classified as producers, 9 as consumers, and 18 as general interest members.

The officers of the committee elected at the meeting on April 4, 1950, for the ensuing term of two years are as follows:

Chairman, A. G. H. Dietz.
Vice-Chairman, D. G. Reid.
Secretary, E. C. Hartmann.
Membership Secretary, R. A. Biggs.

The subcommittee organization remains unchanged with the exception of one change among chairmen; J. J. Lamb is the new chairman of Subcommittee IV, replacing J. M. Stevens.

Committee C-19 sponsored a session at the San Francisco Meeting on October 13, 1949, at which time the following papers were presented:

"Preliminary Considerations for Testing Sandwich Radome Materials,"[2] by G. R. Huisman and R. H. Wight,
"Testing of Sandwich Constructions at the Forest Products Laboratory,"[3] by Edward W. Kuenzi, and
"Durability Tests of Metalite Sandwich Construction,"[4] by David G. Reid.

One of the main activities of Committee C-19 is, of course, the development of test methods applicable to structural sandwich construction. There has been a considerable need and demand for such standardized test methods on the part of both producers and consumers. During the year, nine tentatives have been prepared as indicated in the subcommittee reports.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties of Basic Materials (R. C. Platow, chairman).—This subcommittee has prepared three proposed tentatives covering the following subjects: mechanical test of core materials, apparent and absolute density, and water absorption, which are now receiving consideration. The subcommittee has agreed to adopt the existing method of test for thermal conductivity by means of the guarded hot plate, as applicable to core materials for sandwich construction.

Subcommittee II on Mechanical Properties of Basic Sandwich Construction (E. W. Kuenzi, chairman).—The subcommittee spent most of the year preparing six tentative methods of testing sandwich construction on the following subjects: compression flatwise, compression edgewise, bending flatwise, shear flatwise, shear edgewise, tension flatwise. All are undergoing revision prior to submission to letter ballot.

Subcommittee on Permanence, Durability and Simulated Service (D. G. Reid, chairman).—The subcommittee has been making an attempt to arrange for exposure tests of sandwich constructions under the leadership of R. A. Biggs of the section on Exposure Facilities. The question of exposure tests procedures is also under consideration. Preliminary arrangements have been worked out for round-robin tests on fatigue of sandwich constructions under the leadership of R. C. Platow.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.
[2] ASTM BULLETIN, No. 164, February, 1950, p. 19.
[3] ASTM BULLETIN, No. 164, February, 1950, p. 21.
[4] ASTM BULLETIN, No. 164, February, 1950, p 28.

Subcommittee IV on Nomenclature and Definitions (J. J. Lamb, chairman).—Subcommittee IV has submitted definitions of sandwich constructions and components and some terms relating to adhesives. The subcommittee is reviewing the definitions adopted by Committee D-14 on Adhesives to determine which are pertinent to sandwich constructions.

This report has been submitted to letter ballot of the committee, which consists of 47 voting members; 32 returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. G. H. DIETZ,
Chairman.

E. C. HARTMANN,
Secretary.

REPORT OF COMMITTEE C-21
ON
CERAMIC WHITEWARES[1]

Since its inception in 1948, Committee C-21 has held three meetings: on October 1, 1948, September 22, 1949, and March 1, 1950. In addition, the Executive Subcommittee has held five meetings. The committee has, at present, a voting membership of 78. In addition to the Executive Subcommittee, the organization comprises a Subcommittee on Nomenclature, a Subcommittee on Tests and Specifications, and a Subcommittee on Research.

Reelected for the ensuing term of two years are the following officers:

Chairman, J. H. Koenig.
Vice Chairman, J. R. Beam.
Secretary, R. F. Geller.

NEW TENTATIVE

The committee recommends that the Proposed Definitions and Terms, developed in Subcommittee I on Nomenclature and approved by letter ballot of the committee, be accepted for publication as tentative, as appended hereto.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman).—This subcommittee developed and submitted to the committee a total of 31 terms and definitions dealing with ceramic ware, ceramic coatings, properties, and general terms. Consideration is now being given to a list of definitions covering additional terms used in the ceramic whitewares field.

Subcommittee II on Tests and Specifications (F. P. Hall, chairman).—This subcommittee has been divided into the following:

Section A on Raw Materials with Subsection A1 on Clays (T. A. Klinefelter, chairman) and Subsection A2 on Nonplastics (R. F. Sherwood, chairman).

Section B on Process Controls (C. H. Commons, chairman).

Section C on Product Tests and Specifications with Subsection C1 on Fundamental Properties (W. C. Mohr, chairman) and Subsection C2 on Products Tests and Specifications, which is inactive.

Subsection C1 has been very active and, as a result of numerous meetings, is now activating a round-robin series of absorption tests. As a result of these tests, the section should be in a position within the next year to make recommendations regarding the method for determining absorption to be used in conjunction with limits referred to in the definitions of terms, referred to earlier in this report.

Subcommittee III on Research (C. J. Harman, chairman).—This subcommittee has been chiefly concerned with considering ways and means of accomplishing the research required by Committee C-21. Committee members have been keeping abreast of technical developments and publications that might have a bearing on the committee's needs.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] These Definitions were accepted as tentative and appear in the 1950 Supplement to ASTM Book of Standards, Part 3.

This report has been submitted to letter ballot of the committee, which consists of 78 voting members; 57 members returned their ballots, of whom 56 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. H. KOENIG,
Chairman.

R. F. GELLER,
Secretary.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL[1]

The organization meeting of Committee C-22 On Porcelain Enamel was held on June 17, 1949, at A.S.T.M. Headquarters in Philadelphia. Interim officers, including chairman, vice chairman, and secretary, and seven executive subcommittee members were elected to serve until the next regular election of officers.

The permanent organization of the committee, including scope and methods of operation, was thoroughly discussed and the Executive Subcommittee directed to take the necessary steps in the preparation of by-laws for the committee and in the organization thereof. This work was completed and the results unanimously accepted by letter ballot by the members well in advance of Spring Committee Week.

On March 1 and March 2, 1950, the committee met in Pittsburgh, Pa. At this meeting, officers for the ensuing two years were elected and the organization of the authorized subcommittees and sections was completed. Committee C-22 at present consists of 38 members, of whom 36 are voting members, with 20 classified as producers, 5 as consumers, and 11 as general interest members.

The election of officers for the ensuing term resulted in the selection of the following:

Chairman, W. N. Harrison.
Vice-Chairman, D. G. Bennett.
Secretary, G. H. Spencer-Strong.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (G. H. McIntyre, chairman).—The subcommittee considered 30 possible research projects which had been suggested by the membership prior to the meeting. Six of the suggested projects were selected for immediate study and members of the subcommittee were assigned to investigate the problems and to report on same at the next meeting of the subcommittee.

Subcommittee II on Nomenclature (F. A. Petersen, chairman).—This subcommittee has taken advantage of the work of the Committee on Classification, Nomenclature and Glossary of the Enamel Division of The American Ceramic Society which recently published a glossary of the porcelain enamel art, using it as a basis for an ASTM tentative. At the same time, the subcommittee will investigate the possibility of preparing a glossary of trade names covering proprietary compounds used in the porcelain enamel industry.

Subcommittee III on Tests and Specifications (R. F. Bisbee, chairman).—By action of the subcommittee, Section 1 on Raw Materials (W. A. Deringer, chairman) and Section 2 on Materials in Process (S. E. Hemsteger, chairman) were authorized to operate jointly as a single section during the preliminary phases of their work. The subcommittee agreed that for the purposes of its work semi-finished materials, such as frit, be considered as raw materials.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

Sections 1 and 2 are compiling a list of existing test methods for the raw materials and materials in process used in the porcelain enamel industry. A comprehensive review will be prepared.

Section 3 on Finished Products (J. H. Gregory, chairman).—This section is investigating available test methods now in use in evaluating 11 major physical properties of porcelain enamel coatings on finished products.

This report has been submitted to letter ballot of the committee, which consists of 32 voting members; 24 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE D 1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS[1]

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: in Atlantic City, N. J., June 26 to 29, 1949, in connection with the Annual Meeting of the Society, and in Pittsburgh, Pa., February 27 to March 1, 1950, in connection with the Spring Group Committee Meetings.

At the June, 1949, meeting, the following three papers were presented: "Direct Reading Attachment for Stormer Viscosimeter,"[2] by John H. Calbeck; "A Method for the Standardization of Krebs Modified Stormer Viscometers,"[3] by C. F. Jackson and W. H. Madson; and "Correlation of Accelerated Weathering Machines," by Roy W. Hill, George Cook, and William E. Moyer under the direction of Arthur W. Van Heuckeroth.

At the March, 1950, meeting, a panel discussion was held on "Colorimetry." Walter C. Granville served as moderator and the panel was composed of E. J. Dunn, Jr., R. S. Hunter, A. E. Jacobsen, M. P. Morse, and F. Scofield. The topics covered in the discussion of the relation between colorimetry and paint were: (1) control of working color standards for visual comparison; (2) accuracy of color measurements made by colorimeters, spectrophotometers, and visual comparison to material color standards; (3) a comparison between visual and instrumental conditions for illuminating and viewing samples.

During the past year, a new Subcommittee on Printing Inks under the chairmanship of P. J. Thoma, and a new Subcommittee on Flash Point under the chairmanship of N. C. Schultze have been organized.

The D-1 Membership Committee, which was organized in 1946 to obtain new members for Committee D-1, particularly members engaged directly in the protective coating industry, has been active. The Membership Committee has succeeded in adding fourteen new members to Committee D-1.

The D-1 Inter-committee Relations Committee, which is composed of the D-1 representatives on other technical committees, is continuing to follow closely the activities of other technical committees which may be of interest to Committee D-1.

Cooperative work with the Federation of Paint and Varnish Production Clubs has continued active during the year under the Joint Committee (M. Van Loo, chairman).

A Special Subcommittee on Location of Exposure Test Sites has been cooperating with the A.S.T.M. Advisory Committee on Corrosion in considering various types of test sites for exposure tests. The committee is continuing to study the dirt collection conditions of certain sites by exposing painted panels.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the recommendation that the Method of Test for Distillation Range of Lacquer Solvents and Diluents be published as tentative. This recommendation was accepted by the Standards Committee on September 22, 1949, and the new tentative method appears in the 1949 Book of A.S.T.M. Standards, Parts 4 and 5, bearing the designation D 1078 - 49 T.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] ASTM BULLETIN, No. 161, October, 1949, p. 33 (TP177).

[3] *Ibid*, p. 36.

NEW TENTATIVES

The committee recommends for publication as tentative the following one new specification and seven methods of test, as appended hereto.[4]

Specifications for:

Pure Para Red Toner, as a tentative revision of Standard Specifications D 475 - 49,[5]

Methods of Test for:

Heptane Number of Hydrocarbon Solvents,
Kauri-Butanol Value of Hydrocarbon Solvents,
Nitrocellulose Diluting Power of Hydrocarbon Solvents,
Chemical Analysis of Blue Pigments,
Chemical Analysis of Chromium Pigments, as a revision of and to replace Standard Methods D 126 - 36,[5]
Chemical Analysis of Zinc Dust, as a revision of and to replace Standard Methods D 521 - 40,[5] and
Varnishes, as a revision of and to be added to Standard Methods D 154 - 47.[5]

REVISIONS OF TENTATIVES

The committee recommends that the following two tentatives be revised as indicated:

Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 46 T),[5] revise as appended hereto.[6]

Tentative Specifications for Venetian Red (D 767 - 48 T),[5] revise as follows:

Section 2 (*a*).—In the table in this section change the requirements for two items as follows:

	Present	Recommended
Moisture and other volatile matter, max., per cent:		
Type I	4.0	4.0
Type II	4.0	6.0
Type III	4.0	8.0

	Present	Recommended
Loss on ignition (at 1500 to 1600 F.), max., per cent:		
Type I	1.0	1.5
Type II	1.0	2.5
Type III	1.0	6.0

Section 2 (*b*).—Change the requirements for moisture by distillation as follows:

	Present	Recommended
Moisture by distillation, max., per cent:[a]		
Type I	2.0	3.0
Type II	2.0	4.5
Type III	2.0	6.0

[a] Expressed as percentage by weight.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following three standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting so that these revisions may be submitted to letter ballot of the Society:

Standard Methods of Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese (D 50 - 36),[5] revised as appended hereto.[6]

Standard Methods of Testing Varnishes (D 154 - 47),[5] revision of the viscosity test as appended hereto.[6]

Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 47),[5] revised by addition of the following definition:

> "Oil color.—An oil paint containing a high concentration of colored pigment, commonly used for tinting paint."

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision[5] of the Standard Specifications and Tests for Soluble Nitrocellulose (D 301 - 48) published since June, 1946, and which involve changes in Sections 5 to 9, be approved for reference to letter ballot of the Society for adoption as standard.

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[4] The new specifications and methods were accepted by the Society and appear in the 1950 Supplement to ASTM Book of Standards, Part 4.

[5] 1949 Book of A.S.T.M. Standards, Part 4

[6] The revised methods were accepted by the Society and appear in the 1950 Supplement to ASTM Book of Standards, Part 4.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends the following tentative revisions in two standards:

Standard Methods of Testing Drying Oils (D 555 - 47),[5] comprising a new test for hydroxyl value and break as appended hereto.[6]

Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 47),[5] comprising the following new definitions of four terms:

Spar Varnish.—A varnish for exterior surfaces. The name originated from its use on spars of ships.

Sealer.—A liquid composition to prevent excessive absorption of finish coats into porous surfaces; also a composition to prevent bleeding (see Size).

Primer.—The first of two or more coats of a paint, varnish, or lacquer system.

Size.—Usually a liquid composition to prevent excessive absorption of wall paints into plaster, old wall paint, and similar porous surfaces; also a liquid composition used as a first coat on metal to improve adhesion at succeeding coats (latter usage is limited to the metal decorating industry).

NOTE.—The terms *sealer* and *size* are almost synonomous, but usage has established certain differences. A *sealer* is ordinarily a thin varnish or clear lacquer and is usually applied on wood and metal surfaces. Ordinary painter's *size* is a thin solution of glue, starch or other water-soluble substance and is usually applied on plaster surfaces, but *size* used in metal decorating is a thin varnish.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends the adoption as standard of the following two tentatives without revision:

Tentative Methods of Testing Ethylcellulose (D 914 - 47),[5] and

Tentative Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 - 48 T).[5]

WITHDRAWAL OF STANDARDS

The committee recommends that the following two standards be withdrawn as they are being revised and replaced by new methods of chemical analysis as mentioned earlier in this report.

Standard Methods of Chemical Analysis of:

Yellow and Orange Pigments Containing Chromium Compounds, Blue Pigments, and Chrome Green (D 126 - 36),[5] and

Zinc Dust (D 521 - 40).[5]

EDITORIAL CHANGES IN TENTATIVES

The committee recommends the following editorial changes in two tentatives:

Tentative Specifications for Red and Brown Iron Oxide Pigments (D 84 - 48 T)[5] changing the description in Section 1 (*a*) of Class I from "Iron oxides commercially pure (including Indian Red)" to read "Commercially pure iron oxides produced by roasting iron salts or iron precipitate."

Tentative Method of Test for Night Visibility of Traffic Paints (D 1011 - 49 T),[5] adding to the present note in Section 3 the following four sentences: "A directional reading portable photoelectric photometer has also been described by Hill and Ecker* which can be used in the daylight for evaluating brightness of highway center line stripes. The angle of incidence and the angle of reflectance somewhat differ from that presented in Method D 1011. This instrument is a photoelectric photometer with a direct reading scale. The instrument is self-contained and employs a small 6-v. battery." The footnote reference to this addition should read as follows: "* J. M. Hill and H. W. Ecker, 'A Direct Reading Portable Photoelectric Photometer for Determining Reflectance of Highway Centerlines,' ASTM BULLETIN, No. 159, July, 1949, p. 69."

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Drying Oils (Francis Scofield, chairman) prepared the new method for hydroxyl value and break test, appended hereto.[6] The subcommittee is conducting cooperative work on diene value, a modified foots test. Work is also being done on the determination of conjugation in drying oils.

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Bituminous Emulsions (E. H. Berger, chairman) is considering revisions of the methods of testing bituminous emulsions. Work is being continued on two specifications for emulsions of the coal-tar and asphalt types, which will be based almost entirely on performance requirements.

Subcommittee IV on Traffic Paint (W. G. Vannoy, chairman) is studying tests for the durability of traffic paints, properties of glass beads, and an accelerated suspension test for traffic paint.

Subcommittee V on Volatile Solvents for Organic Protective Coatings (M. B. Chittick, chairman) prepared the new methods of test for heptane number, Kauri-butanol value, and nitrocellulose diluting power of hydrocarbon solvents, as appended to this report.[4] The subcommittee has under development methods of test for naphtha tolerance, viscosity reduction, and copper corrosion.

Subcommittee VI on Definitions (C. H. Rose, chairman) prepared definitions for spar varnish, primer, size, and oil color.

Subcommittee VII on Accelerated Tests (R. H. Sawyer, chairman) prepared a revision of the Tentative Method of Preparation of Steel Panels for Testing Paint, Varnish, Lacquer, and Related Products (D 609 - 46 T). It is preparing a revision of the existing standard for wood panels, D 358 - 48 T, and is continuing cooperation tests on humidity and immersion exposure testing.

Subcommittee VIII on Chemical Analysis (C. Y. Hopkins, chairman) submits for immediate adoption a revision in the Standard Methods of Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese (D 50 - 36). It has also prepared three new Tentative Methods of Chemical Analysis of: Yellow, Orange, and Green Pigments Containing Lead Chromate, and Chromium Oxide Green (to replace Methods D 126 - 36); Zinc Dust (Metallic Powder) (to replace D 521 - 40); and Blue Pigments.

The subcommittee is engaged in preparing a new method of test which will include general tests applicable to pigments, such as water soluble content, hydrogen ion concentration, acidity or alkalinity by titration, etc. Work is also being conducted on the analysis of white lead, titanium dioxide, pigments treated with additive agents, and aluminum pigments.

Subcommittee IX on Varnishes (J. C. Weaver, chairman) prepared a tentative revision of the Standard Methods of Testing Varnishes (D 154 - 47) to consist of the addition of methods of test for "dust free" as determined by the use of cotton linters, "through dry" (thumb pressure) and "tack free" (Zapon Tester) and elasticity and toughness. A revision of the viscosity test in Methods D 154 is recommended for immediate adoption. Cooperative tests are being conducted on eight different samples of varnish to perfect a rapid means of determining non-volatile matter.

A general paper has been prepared covering the numerical relationship between flow times of various efflux cups used in the coatings industry, conversion formulas for these cups, Ford cup standardization, a survey of preference for methods of viscosity measurements and certain corrections of details on the use of Gardner-Holdt tubes.

Surface tension is an important variable in bubble viscometers and a study of the exact relationship of surface tension to viscosity measurement in Interchemical and Gardner-Holdt bubbles is recommended as a research project worthy of sponsorship by the coatings industry.

Work is being done on a method for

determining viscosity by means of the Ford type viscosity cups, based on the work of the Chicago Paint and Varnish Production Club. Further work on bubble tube or other types of viscometers applicable to varnishes and related materials is being conducted.

A group studying alkali resistance has analyzed 18 questions in a poll of Subcommittee IX and has developed from this a further revision of the sections in Methods D 154. This revision will be evaluated in cooperative testings of several representative varnishes. Water resistance may be studied in similar fashion at a later date.

A group on exterior durability of varnish is beginning a cooperative study of five varnishes on wood and metal panels for exposure by several individuals in several parts of the country. Methods of application and uniform observation and reporting will be given close attention.

A new group has been appointed to follow the activities of the New York Production Club in developing a print-free test. A group on color of transparent liquids has been formed to cooperate with other groups including the Inter-Society Color Council in replacing or revising the 1933 Gardner Color Standards to obtain more acceptable color standards of greater permanency.

Subcommittee X on Optical Properties (W. C. Granville, chairman) is continuing to study the problem of developing methods of test for absolute hiding power and small color differences. For better discrimination of low-gloss finishes, the development of a method of test for 85-deg. sheen is being considered.

Subcommittee XI on Resins (C. T. Pickett, chairman) is preparing a proposed method of test for total chlorine in resins for surface coatings which is near completion.

Round-robin cooperative tests are in progress on the following: Precipitation method for total solids, methods for determination of softening point, solvent tolerance of amine resins, and Swann method of determination of phthalic anhydride in the presence of other unsaturated acids.

Surveys are in progress on the determination of specific gravity, clarity and color of solid resins; chemical and physical identification of phenolic resins and vinyl polymers and copolymers.

Subcommittee XII on Exterior Exposure Testing of Paints on Wood (Wayne Fuller, chairman) is preparing a revision of the Tentative Method of Conducting Exterior Exposure Tests of Paints on Wood (D 1006 - 49 T). It is anticipated that this revision will be ready for submission to Committee D-1 at the June meeting. Forms for the recording of exposure data are being developed in collaboration with the Federation of Paint and Varnish Production Clubs.

Subcommittee XIII on Shellac (C. C. Hartman, chairman) met jointly with collaborators from the lac industry and the Federal Government on October 18, 1949, at the American Standards Association Headquarters in New York City. This group, functioning as the American Committee on the International Standards Organization Technical Committte 50 on Shellac, considered proposed Indian Standard Specifications for Seedlac, Shellac and Dry Bleached Shellac, respectively. The comments and suggested changes on these specifications, as approved by the committee, were transmitted in writing through the ASA to the Indian Secretariat for consideration by the International Committee which met in New Delhi, January 16 to 19, 1950.

Subcommittee XIV on the Electrometric Testing of Paint Films (William Shaw, chairman) has been preparing a method for conducting time potential tests on paint films. This proposed method is being studied by a working group of the subcommittee which is conducting cooperative tests.

Subcommittee XV on Pigments (C. L. Crockett, chairman) has prepared revisions of the Standard Specifications for Pure Para Red Toner, Light (D 475 - 49) and Tentative Specifications for Venetian Red (D 767 - 48 T), also editorial changes in the Tentative Specifications for Red and Brown Iron Oxide Pigments (D 84 - 48 T). It is preparing a new specification for calcium carbonate and a revision of the Standard Specifications for Magnesium Silicate Pigment (D 717 - 45).

Consideration is also being given to the revision of some of the iron oxide pigment specifications with particular reference to changing the calcium carbonate content in venetian red and adding a requirement for maximum water soluble matter in some of the other iron oxide specifications where this is not already specified.

Subcommittee XVI on Printing Inks (Paul J. Thoma, chairman) is a new subcommittee and has seven groups in operation under group chairmen as follows:

Rubproofness, C. A. Blessing,
Fineness of Grind, J. C. Braznell,
Drying Time, E. F. Carman,
Printing Ink Rheology, M. C. Rogers,
Methods Review, R. H. Simmons,
Definitions, W. C. Walker, and
Paper-Ink Relationship, M. Zucker.

Subcommittee XVII on Flash Point (N. C. Schultze, chairman) is also a new subcommittee which was established to study and compare methods for the determination of flash point, and establish one or more standard methods, suitable for the determination of the flash point of paints, varnishes, lacquers, and related products and materials used in the manufacture thereof. The subcommittee will first consider a method based upon the Tag open cup tester.

Subcommittee XVIII on Physical Properties (M. R. Euverard, chairman) has ten active groups working on the following subjects:

Oil absorption of pigments,
Application of uniform films,
Consistency of pastes,
Adhesion,
Dry film thickness,
Specific gravity of pigments,
Hardness of paint films,
Chalk resistance of titanium pigments,
Permeability of paint films, and
Fire retardency of paints.

New groups have been formed to study wet film thickness and fineness of grind.

Subcommittee XXV on Cellulose and Related Products (F. H. Lang, chairman) recommends the adoption as standard of the tentative revision of Specifications and Tests for Soluble Nitrocellulose (D 301 - 48) and adoption as standard of the Tentative Methods of Testing Ethylcellulose (D 914 - 47 T). The subcommittee is preparing specifications for methyl isobutyl ketone and for methanol, and a method of test for purity of acetone and methylethyl ketone. The Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 49) are being studied as regards the possible separation of a number of test procedures from this method and to issue them as separate methods.

Subcommittee XXIX on the Painting of Metals (A. J. Eickhoff, chairman) is starting a project on the preparation of standards for ferrous surfaces for painting. This may be in the form of a combined written-pictorial report. When this work is completed, the subcommittee plans to study the qualifications of paints applicable to the different surfaces. A project is also being started to investigate the painting of aluminum and its alloys.

This report has been submitted to letter ballot vote of the committee, which consists of 334 voting members; 105 members returned their ballots, of whom 85 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

C. H. ROSE,
Secretary.

REPORT OF COMMITTEE D-2

ON

PETROLEUM PRODUCTS AND LUBRICANTS[1]

Since the Annual Meeting in 1949, Committee D-2 on Petroleum Products and Lubricants held a committee week October 10 to 14, 1949, and a regular meeting February 20 to 24, 1950. The committee week was held in San Francisco in conjunction with the first Pacific Area National Meeting of the Society. In addition to the regular meeting in Washington, D. C., in February, Committee D-2 and its subcommittees met in Atlantic City, June 26 to 30, 1950.

The voting membership of the Committee is currently 93, of whom 43 are classified as Producers, 35 as Consumers, and 15 as General Interest members.

Committee D-2 announces with regret that C. Dantsizen found it necessary to resign the chairmanship in August, 1949, for reasons of health. Mr. Dantsizen has served as chairman since 1946.

During the year, the committee suffered loss of four of its members of long standing: Eugene A. Ayres, because of the assumption of other responsibilities within his company; R. R. Matthews and E. B. Phillips, through retirement from active duty in their respective companies; and H. C. Dickinson, deceased. Dr. Dickinson's death was mourned throughout the committee.

The committee held its 28th Annual D-2 Dinner on Tuesday evening, June 27, 1950, at which two of its most active members, A. E. Miller and C. A. Neusbaum, were guests of honor.

Reorganization of Committee D-2

The reorganization of Committee D-2 which has been under way for the past three years is now approximately complete. The standing subcommittees of Committee D-2 include, at present, 11 Technical Committees, 11 Research Divisions, 5 Special Subcommittees, and 5 Coordinating Divisions. These standing subcommittees are further subdivided into approximately 125 working groups or sections. Later in this report, under Activities of Standing Subcommittees, a statement is included after the listing of each newly formed committee or division indicating its former position, if any, in the committee structure.

Newly authorized during the past year are Technical Committee M on Petroleum Wax, under the chairmanship of A. M. Heald of the Marathon Corp., which will function as a Joint TAPPI-ASTM Committee on Petroleum Wax; and Research Division XI on Calorimetry. The former group has already held its organization meeting. The latter group is still in the process of organization.

Section I on Oil Turbine Systems of Technical Committee C has also been reorganized as a Joint ASTM-ASME Committee on Steam Turbine Lubrication.

Recommendations Accepted by the Administrative Committee on Standards

On September 22, 1949, the Administrative Committee on Standards accepted a recommendation of Committee D-2 that the Tentative Method of Test for Chlorine in Lubricating Oils by the Bomb Method (D 808 - 44 T) be revised. The revised method, D 808 - 49 T, appears in the 1949 Book of A.S.T.M. Standards, Part 5, and in the 1949 Compilation of A.S.T.M. Standards on Petroleum Products and Lubricants.

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.**

On March 1, 1950, the Administrative Committee on Standards accepted the recommendation of Committee D-2 that the scope of the Tentative Method of Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 - 49 T) be revised. The revised method appears in the 1949 Book of A.S.T.M. Standards, Part 5, and in the 1950 Appendix to the ASTM Manual of Engine Test Methods for Rating Fuels.

On May 25, 1950, Committee D-2 submitted to the Administrative Committee on Standards recommendations for the following four new tentative methods and a revision of an existing tentative:

New Tentative Methods of:

Gaging Petroleum and Petroleum Products
Sampling Petroleum and Petroleum Products
Measuring the Temperature of Petroleum and Petroleum Products
Volume Calculations and Corrections

Revision of Tentative Method of:

Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 - 47 T)

When the above recommendations are accepted by the Society, the four new methods, and the revised Method D 96 - 50 T, together with other related material, are to be published in a separate manual entitled "A.S.T.M. Manual on Measuring and Sampling Petroleum and its Products," authorization for which was included in the 1949 Annual Report of Committee D-2.

AMERICAN STANDARDS

Since the Annual Meeting of the Society in 1949, the American Standards Association, upon recommendation of Sectional Committee Z11 on Petroleum Products and Lubricants, has approved the following A.S.T.M. Standards as American Standard:

D 217 - 48, Method of Test for Cone Penetration of Lubricating Grease (Z11.3—1949)
D 810 - 48, Methods of Test for Sulfated Residue, Lead, Iron, and Copper, in New and Used Lubricating Oils (Z11.57—1949)
D 811 - 48, Methods of Chemical Analysis for Metals in Lubricating Oils (Z11.56—1949)
D 473 - 48, Method of Test for Sediment in Fuel Oil by Extraction (Z11.58—1949)
D 187 - 49, Method of Test for Burning Quality of Kerosine (Z11.17—1949)
D 664 - 49, Method of Test for Neutralization Value (Acid and Base Numbers) by Electrometric Titration (Z11.59—1949)
D 873 - 49, Method of Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method) (Z11.60—1949)
D 938 - 49, Method of Test for Congealing Point of Pharmaceutical Petrolatums (Z11.61—1949)
D 941 - 49, Method for Measurement of Density of Hydrocarbon Liquids by the Pycnometer (Z11.62—1949)
D 525 - 49, Method of Test for Oxidation Stability of Gasoline (Induction Period Method) (Z11.63—1949)
D 381 - 49, Method of Test for Existent Gum in Gasoline (Air-Jet Evaporation Method) (Z11.36—1949) (revision of Z11.36—1947)
D 323 - 49, Method of Test for Vapor Pressure of Petroleum Products (Reid Method) (Z11-44—1949) (revision of Z11.44—1943)
D 357 - 49, Method of Test for Knock Characteristics of Motor Fuels by the Motor Method (Z11.37—1949)
D 127 - 49, Method of Test for Melting Point of Petrolatum and Microcrystalline Wax (Z11.22—1949) (revision of Z11.22—1932)
D 156 - 49, Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (Z11.35—1949) (revision of Z11.35—1938)
D 129 - 49, Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (Z11.13—1949) (revision of Z11.13—1944)
D 288 - 49, Definitions of Terms Relating to Petroleum (Z11.28—1949)

D 526 - 48 T, Method of Test for Tetraethyllead in Gasoline (Z11.48—1949)

The ASA has also reaffirmed the following American Standards on recommendation of Sectional Committee Z11:

D 88 - 44, Method of Test for Viscosity by Means of the Saybolt Viscosimeter (Z11.2—1944), Reaffirmed 1949

D 565 - 45, Method of Test for Carbonizable Substances in White Mineral Oil (Liquid Petrolatum) (Z11.49—1945), Reaffirmed 1949

D 666 - 44, Method for Conversion of Kinematic Viscosity to Saybolt Furol Viscosity (Z11.53—1944), Reaffirmed 1949

D 612 - 45, Method of Test for Carbonizable Substances in Paraffin Wax (Z11.50—1945), Reaffirmed 1949

ACTIVITIES OF STANDING SUBCOMMITTEES

Technical Committee A on Gasoline (H. M. Smith, chairman) has continued the study of properties of motor fuels and has proposed that the Tentative Specifications for Gasoline (D 439 - 49 T) be revised to raise the 13.5 lb. maximum Reid vapor pressure to 15.0 lb. for the W(winter) fuel. This proposal originated in Section V, Specifications and Significance of Tests.

Section V is currently without a chairman, since the death of H. C. Dickinson, but is continuing the study of climatological data and their relationship to motor fuel volatility as those data are made available by the Weather Bureau. A study of the Bureau of Mines National Motor-Gasoline Survey for the summer of 1949 indicates that the present minimum octane limits given in Specifications D 439 - 49 T conform closely to the minimum values shown by the survey for both regular and premium grades. Section V will continue to observe trends in gasoline characteristics as revealed by the surveys and will revise the specifications when necessary. This section has called attention to the fact that the discontinuance of the Standard Method of Test for Detection of Free and Corrosive Sulfur Compounds in Gasoline (D 130 - 30) and the substitution of D 130 - 49 T, Tentative Method of Test for Free and Corrosive Sulfur in Petroleum Products, will require editorial revision of Specifications D 439.

Technical Committee B on Lubricating Oils (W. S. James, chairman) sponsored an A.S.T.M. Symposium on High-Additive Content Lubricating Oils on October 12, 1949, during the Pacific Area National Meeting in San Francisco. The following four papers were presented:

"Does Service Performance Justify Higher Quality Lubricating Oils," by W. B. Bassett, Lubri-Zol Corp.

"High Detergency Motor Oils," by A. B. Boehm, Enjay Co., New York, and C. O. Tongberg, Standard Oil Development Co.

"Five Years' Experience With Special Heavy-Duty Oils," by R. E. Jeffrey and J. A. Edgar, Shell Oil Co., Inc.

"Possible Future Military Engine Oils," by N. L. Klein, Office of Chief of Ordnance, Dept. of the Army.

These papers have been made available in a special technical publication by the Society.[2]

The committee heard a paper on "Experiences in the Lubrication of Ordnance Instruments," by Henry Gisser of the Frankford Arsenal at a meeting on February 23, 1950, of Section U-V on Instrument Oils (E. H. Erck, chairman). On the same date, at the meeting of Section U-VI on Lubricants for Air Compressors and Compressed Air Tools (C. M. Larson, chairman) the committee heard a paper on "Types of Compressors and the Behavior of Additives," by K. L. Hollister of The Texas Company.[3]

Two future symposia are planned, one for February, 1951, again on High-Additive Content Oils; the other for June, 1951, on Lubrication of Extreme-Pressure Conditions. Other groups within Committee D-2 will probably join with Technical Committee B in the sponsorship of this latter symposium.

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[2] Issued as a separate publication, *STP No. 102*, June, 1950.

[3] Published in ASTM BULLETIN, No. 170, December, 1950, p. 35 (TP261).

Upon the recommendation of Section Q-I on Physical and Chemical Changes (L. L. Davis, chairman), Technical Committee B has proposed that the Method of Test for Sulfated Residue from New Lubricating Oils (D 874 - 47 T), be continued as tentative. A study of methods for measuring the dilution of crankcase oils from both gasoline and diesel engines is also under way in Section Q-I.

Section Q-II on Oxidation and Bearing Corrosion (H. R. Wolf, chairman) is currently studying a proposed method for measuring corrosion by means of lead strips.

Technical Committee B continued to serve as a forum for the discussion of proposed revisions in the SAE classification of motor oils. Several reports from the Special Committee on Classification of Motor Oils (J. L. McCloud, chairman) have been heard and consideration has been given to several additional recommendations from interested individuals and groups.

Technical Committee C on Turbine Oils (F. C. Linn, chairman) was responsible for a technical session of the Society, held in San Francisco, October 12, 1949. This Symposium on Turbine Oils included four papers:

"Lubrication Problems and Requirements of Gas Turbine Equipment," by F. C. Linn, General Electric Co.

"Operating Experience and Problems Relative to Lubricating Systems of Steam Turbine Sets for Utilities," by V. Estcourt, Pacific Gas and Electric Co.

"Specific Problems Pertaining to Lubrication of Industrial Turbines," by A. S. Morrow, Shell Oil Co., Inc.

"Lubrication of Marine Turbine Propulsion Equipment," by F. S. Jones, Socony-Vacuum Oil Co., Inc.

These papers are being published as a technical pamphlet by the Society.[4]

Section I on Oil Systems of Turbines has been given a dual status and made an ASTM-ASME Joint Committee on Steam Turbine Lubrication. It has completed its work on two new recommended practices: a Proposed Recommended Practice for Cleaning Turbine Lubrication Systems After Service, and a Proposed Recommended Practice for the Purification of Turbine Oils. These Recommended Practices will be presented as a paper before the Petroleum Division of the American Society of Mechanical Engineers in New Orleans in September, 1950, and will be published in *Mechanical Engineering*. Section I is continuing to work on a Recommended Practice for the Design of Turbine Lubricating Systems and on the problem of turbine flushing oils and practices.

In its dual capacity this group will continue to serve as Section I of Technical Committee C, and all matters pertaining to test methods will be referred to Technical Committee C for processing in the established manner for publication by the A.S.T.M. Matters appropriate to A.S.M.E. will be channelled to that organization.

Technical Committee C has approved and passed on to Committee D-2 a revision of the Method of Test for Rust-Preventing Characteristics of Steam Turbine Oils in the Presence of Water (D 665 - 49 T), which was worked out by Section II on Rusting (W. P. Kuebler, chairman).

Section III on Oxidation of Turbine Oils (E. A. Reehl, chairman) has called attention to a number of minor editorial revisions that were necessary in the Method of Test for Oxidation Characteristics of Steam Turbine Oils (D 943 - 47 T), and initiated the proposal of

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[4] Issued as a separate publication *STP No. 105*, September, 1950.

Technical Committee C that this method be continued as tentative.

Section IV on Emulsion Tests (M. D. Baker, chairman) is working currently on a new test method for evaluating the emulsifying tendencies of turbine oils. This section initiated the proposal of Technical Committee C that the Tentative Method of Test for Interfacial Tension of Oil Against Water by the Ring Method (D 971 – 48 T) be adopted as standard.

Section V on Film Strength Testing (C.D. Wilson, chairman) is carrying out cooperative work with the A.S.M.E. Research Committee on Lubrication, involving bench testing of oils of varying pressure-viscosity characteristics.

Section VI on Compatibility Testing (W. C. Witham, chairman), which was organized during the past year, is initiating a preliminary cooperative program for determining the effects resulting from the mixing of two or more types of turbine oil.

Technical Committee E on Burner Fuel Oils (M. A. Powers, chairman) is continuing to follow the current practices in the field of burner fuel oils. This committee has agreed to editorial changes in the Specifications for Fuel Oils (D 396 – 48 T), to make these specifications conform to the new Tentative Method of Test for Free and Corrosive Sulfur in Petroleum Products (D 130 – 49 T).

Technical Committee F on Diesel Fuels (C. G. A. Rosen, chairman) held a Symposium on the Effect of Fuel Upon Diesel Engine Deposits in San Francisco on October 13, 1949. The following five papers were delivered:

"Influence of Diesel Fuels on Engine Deposits," by H. F. Bryan of International Harvester Co.

"Fuels for Diesel Engines—A Review," by C. C. Moore and W. L. Kent of the Union Oil Co. of California.

"The Challenge of the Future Diesel," by C. G. A. Rosen of the Caterpillar Tractor Co.

"Diesel Fuels and Engine Deposits," by J. T. Koot and A. G. Cattaneo of Shell Development Co.

"Effect of Fuel Upon Diesel Engine Deposits," by V. M. Stanich of General Petroleum Corp.

It also sponsored an informal discussion on railway diesel fuels in February, to which representatives of the railroads and locomotive builders were invited. As a result of the discussion, Technical Committee F will organize a new Section III which will act as a liaison group with the railroads, cooperating specifically with the Association of American Railroads.

Section I, CRC Liaison (W. J. Backoff, chairman) made regular reports on the current diesel fuel projects which are being carried out by the CRC. These include the following:

1. Effect of fuel sulfur on engine operation.
2. Fuel deterioration, effect on engine performance.
3. Ignition quality testing.
4. Railroad field service testing.
5. Front-end volatility.
6. Corrosive tendencies of fuels and fuel systems.
7. Deposit-forming characteristics.
8. Full-scale test techniques.
9. Combustion characteristics—ignition delay bomb.

Section II on Specifications (H. M. Gadebusch, chairman) has worked out some revisions of the Specifications for Diesel Fuels (D 975 – 49 T) which have been approved by Technical Committee F and referred to Committee D-2. These revisions are being submitted to the Society in this report.

Technical Committee G on Lubricating Greases (R. C. Adams, chairman) has continued to expand its activities and membership during the past year. Fourteen working subsections are now active.

Section I, Subsection 3 on Oxidation Characteristics of Greases has completed a careful restudy of the Method of Test for Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method

(D 942 – 47 T), as a result of which the method is being recommended for adoption as standard, with certain minor revisions.

Section II, Subsection 4 on Flow Characteristics (L. C. Brunstrum, chairman) initiated a proposal that the Method of Test for Apparent Viscosity of Lubricating Greases, which was published as information as Appendix II of the 1949 Report of Committee D-2, be accepted as tentative. No adverse criticism of this method has been received since publication and the method is judged to be in conformance with accepted practice.

Section II, Subsection 1 on Evaporation Test for Greases at High Temperature (F. E. Woodward, chairman) is investigating certain minor difficulties encountered with the use of the Method of Test for Evaporation Loss of Lubricating Greases and Oils (D 972 – 48 T), on oil samples. It is also considering the extension of this method to high temperatures. Pending the completion of this work, Technical Committee G has proposed that Method D 972 be retained as tentative.

Other technical studies on the program of Technical Committee G include:

1. A study of the effect of grease on metals in storage.
2. A study of the Method of Test for Cone Penetration of Lubricating Greases (D 217 – 48) in conjunction with multiple mechanical working of grease samples.
3. A cooperative test program on the determination of dirt in greases.
4. An extensive cooperative test program for evaluation of the wheel bearing grease tester described in the Proposed Method of Test for Performance Characteristics of Wheel Bearing Grease published as Appendix I to the 1948 Report of Committee D-2.[5]
5. The development of test methods for the determination of lead, sulfur, chlorine and phosphorus in greases. Research Division III on Elemental Analysis. (C. M. Gambrill, chairman) is collaborating in this study.
6. The development of methods for determining the consistency of semi-fluid greases.
7. Development of methods for predicting storage stability of greases.
8. Investigation of the micro penetrometer and micro worker.
9. Development of functional tests for high-temperature antifriction bearing greases, wheel-bearing greases, low-temperature greases, and water-resistant greases.
10. A study by section IV on Thixotropy (B. B. Farrington, chairman) of the work softening and age hardening of greases.
11. Development of a standard ball bearing grease testing machine.
12. Development of suitable standardized test equipment for determining the starting torque and running torque characteristics of ball bearing greases at low temperatures.

Committee D-2 has endorsed a proposal of Technical Committee G that the Society sponsor a symposium, at an early date, on the subject of fretting corrosion or friction-oxidation. This problem concerns many technologists in addition to the grease makers and users.

Technical Committee H on Light Hydrocarbons (W. G. Lovell, chairman) has continued its work throughout the past year at a high level of activity. Upon recommendations originating in its various sections, Technical Committee H has proposed three new tentatives as follows:

Method of Test for Boiling Point Range of Polymerization Grade Butadiene, published as Appendix VII to the 1949 Report of Committee D-2.[6]

Tentative Method of Test for Carbonyl Content of Butadiene, appended hereto.[7]

Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases, published as Appendix V to the 1948 Report of Committee D-2,[7a] except that values above 0.600 will be deleted and values for 0.590 and 0.595 revised based on current information and calculations.

Technical Committee H has also proposed that the Tentative Method of

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[5] *Proceedings*, Am. Soc. Testing Mats., Vol. 48, pp. 303 to 308 (1948).

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, pp. 364 to 368 (1949).

[7] The new method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 5.

[7a] *Proceedings*, Am. Soc. Testing Mats., Vol. 48, pp. 318 to 327 (1948).

Test for Determination of Butadiene Content of Polymerization Grade Butadiene (D 973 - 48 T), be adopted as standard.

This committee has also sponsored the compilation of a tabulation of physical constants of hydrocarbons and has proposed that it be published as a miscellaneous technical publication by the Society, in keeping with a main purpose of A.S.T.M., namely "the promotion of knowledge of the materials of engineering." The data in the compilation were gathered from the literature and are not to be construed as A.S.T.M. standards. Committee D-2 has concurred in this recommendation.

Other items on the program of Technical Committee H are as follows:

Section I (formerly Section III) on Chemical Utilization including Pure Hydrocarbons (R. F. Dunbrook, chairman) is continuing its program of development of methods of test for use in connection with butadiene, and is considering methods of test and specifications for pentanes, pentenes, and petroleum hexane for pharmaceutical use.

Section II (formerly Section VI) on Liquefied Petroleum Gas (E. W. Evans, chairman) is cooperating with the California Natural Gasoline Association and the Natural Gasoline Association of America in the study of vapor pressure, sulfur content, and methods of sampling liquefied petroleum gas.

Technical Committee J on Aviation Fuels (J. T. Hendren, chairman) is in the process of revising the Specifications for Aviation Gasoline (D 910 - 48 T), by removing therefrom all descriptions of test procedures. These procedures will either be incorporated in existing methods or will be issued as new tentatives.

Specifically, Technical Committee J is proposing, at this time, the removal from the aviation gasoline specifications of the sections on net heat of combustion, acidity of distillation residue, copper strip corrosion, visible lead precipitate and water tolerance.

The description of the procedure for net heat of combustion is being incorporated in the Method of Test for Thermal Value of Fuel Oil (D 240 - 39); new tentative methods are being submitted covering the procedures for acidity of residue from distillation of gasoline and of petroleum solvents (see Report of Research Division VI), and water tolerance of aircraft fuels. Procedures for corrosion (copper strip) and visible lead precipitate have already been incorporated in the Method of Test for Free and Corrosive Sulfur in Petroleum Products (D 130 - 49 T), and the Method of Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method) (D 873 - 49), respectively. The text of the Specifications for Aviation Gasoline (D 910) will continue, temporarily, to incorporate the procedures for color, gum by copper dish, and freezing point of gasoline. The problem of preparing replacement methods for these three procedures has been referred to Research Division IX on Color, Research Division V on Analysis of Fuels, and Research Division VII on Flow Properties.

Technical Committee K on Cutting Fluids (E. M Kipp, chairman) has succeeded in selecting several test fluids which will be used by both Section I on Laboratory Evaluation of Cutting Fluids (T. G. Roehner, chairman) and Section II on Plant Evaluation of Cutting Fluids (M. E. McKinney, chairman) for the appraisal of various methods for determining physical and chemical characteristics and operational behavior of cutting fluids.

Technical Committee L on Tractor Fuels (E. M. Hughes, chairman) has succeeded in reaching agreement on a definition of tractor fuel and on specification limits for a light and a regular grade. The proposed definition and specifications are included as information in Appendix VIII to this report.

Technical Committee M on Petroleum Wax, which functions as a TAPPI-ASTM Joint Committee on Petroleum Wax, held its organizational meeting on February 20, 1950. Twenty-four producers and twenty-seven consumers and general interests accepted membership on this committee. Officers were elected, as follows: Chairman, A. M. Heald of the Marathon Corp.; Vice-Chairman, R. B. Killingsworth of Socony-Vacuum Oil Co., Inc.; and Secretary, A. P. Anderson of Shell Oil Co., Inc. The scope of this committee will cover methods of test, nomenclature, and specifications for petroleum wax.

Eight sections of this committee have been appointed to work in the following fields: strength testing, flow tests, stability tests, sensory tests, composition, blocking, sealing and laminating, and scuff-gloss. Certain testing programs in this field, already initiated under the sponsorship of the Technical Association of the Pulp and Paper Industry, are being incorporated into the operations of this technical committee.

Research Division I on Combustion Characteristics (D. B. Brooks, chairman) is continuing its active engine testing program. No major changes in the five knock test methods are anticipated this year. The division recommends that the Method of Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 – 48 T) be continued as tentative at this time. In the course of its regular work of keeping close check on the accessories and parts available for the ASTM-CFR Knock Test Engine, the division has inspected the following innovations proposed by the Waukesha Motor Co. and approved the changes as not affecting test results:

1. Improved wiring materials for the panelboard conforming to the Underwriters' code with regard to electrical and insulating characteristics and color used in the various circuits.
2. A curved spark control arm which eliminates interference when a 4-bowl carburetor is used.
3. An improved coolant-return pipe which is more easily removed and installed during engine servicing.
4. A plastic oil-level sight gage for the high-speed crankcase which gives improved oil level visibility.

Active projects which are currently under way in the various sections of this Division include: the packaging and storage of the high-purity *iso*octane and *n*-heptane; the testing of an electronic mixture-heater regulator and a selenium rectifier for possible elimination of the d-c. generator on the ASTM-CFR knock test engine; the redesign of the engine panelboard; the development of a micro method for octane determination on very small samples; redesign of other engine parts; the study of the effect of barometric pressure on the rating of fuels; and a study of the reproducibility of the "F-21" method for rating aviation gasolines.

Division II on Measurement and Sampling (formerly Subcommittee XV) (L. C. Burroughs, chairman) has just completed the initial phase of its project to publish a three-volume set of *Oil Measurement Standards*. The first volume, entitled "A.S.T.M. Manual on Measurement and Sampling of Petroleum and Petroleum Products," has been accepted by the Administrative Committee on Standards[8] and will be published in the near future. It will contain a suitable introduction; four new Tentative Methods on Gaging, Sampling, Volume Calculations and Corrections, and Temperature Measurement; also the Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 – 39) and the Tentative Method of Test for Water and Sediment in Petroleum Products by Means of Centrifuge (D 96 – 50 T).

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[8] Issued as a separate publication.

Considerable progress has been made by Division II and the British Institute of Petroleum in the cooperative preparation of a complete set of Standard Oil Measurement Tables. The initial calculations have been completed, and the current phase involves checking and type setting the resultant information. The calculation was done by Mr. Jack Sherman, of The Texas Company, on IBM automatic calculating machines. The American set of tables, which will be published as Volume II of the A.S.T.M. *Oil Measurement Standards*, will occupy approximately 650 pages, and the British and metric sets about 400 to 500 pages each.

Section E on Tank Calibration (H. C. Packard, chairman) has completed the initial draft on the Calibration of Vertical Cylindrical Tanks, which is designed to become a part of Volume III of the *Oil Measurement Standards*.

Research Division III on Elemental Analysis (C. M. Gambrill, chairman) has been organized to embrace a number of closely affiliated former analytical subcommittees and technical committee sections.

Two symposia were held under the auspices of this division during the past year. The first, held in October, 1949, in San Francisco, covered Modern Chemical and Instrumental Methods for the Determination of Metals in Petroleum Products, with the following six papers:

"Polarographic Determination of Copper, Nickel, and Vanadium in Petroleum Products," by D. R. Hild, P. C. Ross and R. O. Clark, Gulf Research and Development Co.

"Photometric Determination of Iron, Copper, Nickel and Vanadium in Petroleum Products," by R. E. Snyder and R. O. Clark, Gulf Research and Development Co.

"Flame Photometric Determination of Calcium in Lubricating Oils," by L. C. Jones, Shell Oil Research Laboratories

"Amperometric Determination of Iron and Copper in Lubricating Oils," by T. D. Parks and Louis Lykken, Shell Development Co.

"Spectographic Determination of Phosphorus and Metal Additives in Oil Using a Solutions Excitation Attachment," by J. P. Pagliassotti and F. W. Porsche, Standard Oil Co. (Indiana)

"Determination of Inorganic Elements in Oils by X-ray Absorption," by R. C. Vollmar, Standard Oil Co. of California.

The second symposium, held February, 1950, in Washington, D. C., was on the subject of Recent Developments in Instrumental Methods for the Determination of Tetraethyllead in Gasoline, and included the following seven papers:

"Rapid Determination of Tetraethyl Lead in Aviation Gasoline," by J. C. Bailie, W. J. Tancig, W. E. Delaney and V. A. Smith, Standard Oil Co. (Indiana)

"Rapid Polarographic Determination of Tetraethyl Lead in Gasoline," by Kent A. Hansen, Thomas D. Parks and Louis Lykken, Shell Development Co.

"A Direct-Reading Polarograph for the Determination of Tetraethyl Lead in Gasoline," by E. B. Offutt and L. V. Sorg, Standard Oil Co. (Indiana)

"X-ray Methods in the Analysis and Preparation of Leaded Gasoline," by H. A. Liebhafsky and E. H. Winslow, General Electric Co.[9]

"Determination of Tetraethyl Lead in Gasoline by X-ray Absorptiometry," by G. Calingaert and F. W. Lamb of the Ethyl Corporation, H. L. Miller of the University of Michigan, and G. E. Noakes of the University of Toronto

"Monochromatic X-ray Methods for the Determination of Tetraethyl Lead in Gasoline," by H. K. Hughes and F. P. Hochgesang, Socony-Vacuum Laboratories

"X-ray Fluorescence Analysis of Ethyl Fluid in Gasoline," by L. S. Birks, E. J. Brooks, H. Friedman and R. M. Roe, U. S. Naval Research Laboratory

The authors of the papers from both of these symposia will arrange for publication in technical journals of their own choice.

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[9] ASTM BULLETIN, No. 167, July, 1950, p. 67 (TP149).

Committee D-2 is supporting a proposal of Research Division III that a joint Symposium on Flame Photometry be held at the A.S.T.M. Annual Meeting in Atlantic City in 1951, probably under the joint sponsorship of Committee C-1 on Cement and Committee D-2.

Section A on Sulfur (formerly Subcommittee VII) (B. J. Heinrich, chairman) has developed revisions in the Tentative Method of Test for Sulfur in Petroleum Products by the Lamp Gravimetric Method (D 90 – 47 T), and the Method of Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (D 129) which have been approved by the Division and proposed to Committee D-2.

Section B on Chlorine (formerly Section A of Subcommittee XI) (P. C. White, chairman) has developed revisions of the Tentative Method of Test for Chlorine in Lubricating Oils by Bomb Method (D 808 – 49 T) which permit the inclusion of greases in the scope of the method and also improve the procedure for the precipitation of silver chloride. These revisions have been proposed to Committee D-2.

Section C on Phosphorus (formerly Section B of Subcommittee XI) (Harry Levin, chairman) has initiated the proposal of Research Division III that the Proposed Methods of Test for Phosphorus in Lubricating Oils, Lubricating Oil Additives, and Their Concentrates, which was published as Appendix VIII to the 1949 Report of Committee D-2[9a] be published as tentative. If this proposal is accepted, an explanatory note will be included in the Tentative Method of Chemical Analysis for Phosphorus in Lubricating Oils (D 809 – 44 T), indicating that Method D 809 may be superseded in the near future by the new tentative. This method is currently being tested for use with greases.

Section D on Metals (formerly Section D of Subcommittee XI) (W. C. Woelfel, chairman) is currently testing proposed methods for the determination of lead in grease.

Section E on Trace Elements (R. O. Clark, chairman) is making a survey of the analytical instruments available in preparation for the initiation of a cooperative testing program, if and when such a program is organized.

Section F on Tetraethyllead in Gasoline (formerly Section VII of Technical Committee A) (J. B. Rather, Jr., chairman) has initiated the recommendation for the continuation as tentative of the Method of Test for Tetraethyllead in Gasoline (D 526 – 48 T) pending the consideration of non-chemical procedures for inclusion in the method.

Section G on Carbon, Hydrogen, Oxygen and Nitrogen (formerly Section G, Subcommittee XXV) (R. Matteson, chairman) has completed its work on the determination of hydrogen (D 1018 – 49 T) and plans to await further development on the oxygen and nitrogen methods before initiating a program of cooperative work.

Research Division IV on Hydrocarbon Analysis (formerly Subcommittee XXV) (S. S. Kurtz, Jr., chairman) has made a number of proposals, some of which were initiated in the various sections. The proposals are described as follows:

A Method of Test for Hydrocarbon Type Analysis of Diesel Fuels is proposed for publication as information only.

The continuance as tentative of the Method of Test for Olefins and Aromatics in Petroleum Distillates (D 875 – 46 T) is recommended in anticipation of revision during the coming year.

Section B on Determination of Olefinic Unsaturation by Chemical Means (E. T. Scafe, chairman) has revised the two methods for bromine number of petroleum distillates which were published as information in Appendices X and XI to the 1949 Report of Committee D-2; and the Division has proposed reissuance of the revised methods as information.

[9a] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 369 (1949).

Section D on Refractive Index, Dispersion, and Density (H. M. Smith, chairman) has developed two new methods of test: one for the Precise Determination of Refractive Index and Refractive Dispersion, the other for Density of Knock Test Standard Normal Heptane and *Iso*octane by the Pycnometer, which are proposed for publication as information. The first of these methods is the outgrowth of an extended study of the technique of precision refractometry by members of this section. The second method was developed in response to a request by Section E on Reference Materials, of Research Division I.

Section E on Determination of Purity by Freezing Point (F. D. Rossini, Chairman) has made studies which resulted in proposals for revisions in the scope and change of certain of the constants used in the Method of Test for Determination of Purity from Freezing Points (D 1016 - 49 T).

Section F on Spectroscopic Methods for the Analysis of Hydrocarbons (S. S. Kurtz, Jr., chairman) has developed two new methods which the division has proposed for issuance as tentative. These are: Method of Test for Analysis of 60 Octane Number *Iso*octane-Normal Heptane A.S.T.M. Knock-Test Reference Fuel Blends by Infrared Spectrophotometry, and Method of Test for 1,3-Butadiene in C_4 Hydrocarbon Mixtures by Ultraviolet Spectrophotometry. The first of these methods was developed at the request of Research Division I. Data showing the accuracy and precision of the latter method, based on cooperative testing by the Rubber Reserve, is included in Table I.

As a result of studies by Section H on Determination of Miscibility Temperatures (R. L. Nix, chairman), the division has proposed continuance as tentative of the Method of Test for Aniline Points and Mixed Aniline Points of Petroleum Products (D 611 - 47 T). Consideration is being given to revising Method D 611 to include the simple test tube apparatus described in the Method of Test for Aniline Points and Mixed Aniline Points of Hydrocarbon Solvents (D 1012 - 49 T), which was developed under the sponsorship of Committee D-1.

Research Division V on Analysis of Fuels (C. A. Neusbaum, chairman) is newly organized and is made up of the former Section I of Technical Committee A, Section IV of Technical Committee E, and Subcommittee XIV.

Section A on Deterioration Products (E. T. Scafe, chairman) has worked out extensive editorial revision of the Method of Test for Existent Gum in Gasoline (Air-Jet Evaporation Method) (D 381 - 49) which the division has proposed to Committee D-2. The revisions were designed for clarification, and include a more detailed specification for the gum solvent used in the test. Acceptance of this proposal requires revision of Section 5 on gum solvent of the Method of Test for Oxidation Stability of Gasoline (Induction Period Method) (D 525 - 49) and of the Method of Test for Oxidation Stability of Aviation Gasoline (Potential Gum Method) (D 873 - 49) so that the gum solvent in all three methods will remain identical. Section A is currently conducting cooperative work in an effort to develop procedures for the determination of existent gum or nonvolatile residue in gasoline containing dissolved nonvolatile lubricating oil, in jet fuels, and in other fuels boiling above the gasoline range.

Section B on Extraneous Material (R. L. Nix, chairman) has developed an improved specification for the bath used in the Method of Test for Water and Sediment in Petroleum Products by

Means of Centrifuge (D 96 – 47 T), and has worked out a procedure for use in testing samples containing waxy material. The division has proposed these revisions to Committee D-2. Section B is currently working to establish a technique, in conjunction with A.S.T.M. Committee E-1 on Methods of Testing, for the calibration of centrifuge tubes.

TABLE I.—RESULTS OF ANALYTICAL COMMITTEE TEST PROGRAM ON ULTRAVIOLET ANALYSIS OF MIXTURES NOS. 13 TO 20 OF BUTADIENE.

NOTE—All mixtures were analyzed by Method No. LM 2.1.85.

Laboratory Number	Butadiene Mixture							
	Mix 13	Mix 14	Mix 15	Mix 16	Mix 17	Mix 18	Mix 19	Mix 20
13	0.0	0.1	18.7	2.2	1.0	0.0	0.1	2.1
	0.0	0.1	18.7	2.2	1.1	0.0	0.1	2.1
	0.0	0.1	18.7	2.2	1.0	0.0	0.1	2.2
19	......	......	20.5	1.5	0.2[a]	0.0	0.0	......
	......	......	20.5	2.0	0.8	0.0	0.0	......
	......	......	21.0	1.7	0.7	0.0	0.0	......
20	......	......	17.0	2.3	0.9	......	......	......
22	0.0	0.0	16.5	1.6	0.7	......	......	......
	0.0	0.0	16.3	1.6	0.7	......	......	......
	0.0	0.0	16.4	1.6	0.7	......	......	......
25	0.3	0.3	18.9	2.0	1.0	0.1	0.1	......
	0.2	0.4	18.8	2.0	1.0	0.1	0.1	1.9
	0.3	0.5[a]	18.8	2.0	1.0	0.1	0.1	1.9
28	0.1	0.1	17.9	1.8	0.9	0.0	0.1	......
	0.1	0.3	17.9	1.8	0.9	0.0	0.0	......
	0.1	0.2	17.6	1.8	0.9	0.0	0.1	......
37	......	......	17.8	2.2	0.8	......	......	......
	......	......	18.2	2.1	0.9	......	......	......
	......	......	18.9	2.1	0.8	......	......	......
39	0.0	0.1	20.2	2.1	0.9	......	......	1.9
	0.0	0.1	20.2	2.1	0.9	......	......	1.9
	0.0	0.2	20.2	2.1	0.9	......	......	1.9
40	......	......	18.6	1.9	1.0	0.0	0.1	......
	......	......	18.6	1.9	1.0	0.0	0.1	......
	......	......	18.6	1.9	1.0	0.0	0.1	......
Synthetic value, mole per cent	0.0	0.0	18.28	1.80	0.93	0.0	0.0	......
Average value, mole per cent	0.07	0.14	18.62	1.95	0.87	0.02	0.07	1.99
Average – Synthetic	+0.07	+0.14	+0.34	+0.15	−0.06	+0.02	+0.07	......
Standard deviation	0.11	0.12	1.28	0.22	0.11	0.04	0.04	0.11
Probable error	0.08	0.09	0.91	0.16	0.08	0.03	0.03	0.09
Probable error/Avg. × 100	114.29	64.29	4.98	8.21	9.20	150.0	42.86	4.52
No. of analyses	15	14	25	25	24	15	15	8
No. of analyses rejected	0	1	0	0	1	0	0	0
No. of laboratories	5	5	9	9	9	5	5	3

[a] Values rejected in calculating average according to a procedure based on the Pierce-Chauvenet criterion.

Research Division VI on Analysis of Lubricants (H. P. Ferguson, chairman) has an organization parallel to that of Research Division V. It contains sections which formerly functioned as Subcommittees XIII, X, and XXIII.

Upon the recommendation of Section A on Neutralization Number and Saponification (H. P. Ferguson, chairman) the division has proposed the continuation as tentatives of Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (D 94 – 48 T) and Method of Test for Neutralization Value (Acid and Base Numbers) by Color-Indicator Titration (D 974 – 48 T) pending further study during the coming year. The Method of Test for Saponification Number of Petroleum Products by Potentiometric Titration (D 939 – 47 T) is proposed for adoption as standard, with one minor revision. A new Tentative Method for Acidity of Residue from Distillation of Gasoline and of Petroleum Solvents, which includes Section 9 (*g*) of the Specifications for Aviation Gasoline (D 910 – 48 T) (see section on the activities of Technical Committee J earlier in this report) has been proposed.

Upon the recommendation of Section

B on Insoluble Matter (L. L. Davis, chairman), the division has proposed the continuation as tentative of Method of Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils (D 893 - 48 T), with a revision in the scope.

Section C on Carbon Residue (F. I. L. Lawrence, chairman) is continuing the study of the Method of Test for Carbon Residue of Petroleum Products (Ramsbottom Carbon Residue) (D 524 - 42).

Research Division VII on Flow Properties (J. C. Geniesse, chairman) incorporates all of the work on viscosity and similar properties which was formerly carried out by Subcommittees V and XVI and Section Q-IV of Technical Committee B.

Section A on Newtonian Liquids (John Watt, chairman) is working on a revision of the Method of Test for Kinematic Viscosity (D 445 - 46 T). Pending the completion of this work, the method is recommended for continuation as tentative.

Section B on Non-Newtonian Liquids (Orrin Clark, chairman) is currently studying the results of the work that was carried out during the past year under the auspices of the American Petroleum Institute on the measurement of viscosity at high rates of shear. These results were presented to Research Division VII at a symposium held in February, 1949, at which the following two papers were delivered:[10]

"The Preparation, Properties, and Viscosity-Shear Behavior of API Test Oils Nos. 103 and 104," by M. R. Fenske, The Pennsylvania State College

"Viscosity Variation With Temperature and Rate of Shear in Four Lubricating Oils," by Sydney J. Needs, Kingsbury Machine Works, Inc.

Section D on Cloud and Pour Points (J. J. Giammaria, chairman) has developed a Method of Test for Pour Stability Characteristics of Winter-Grade Motor Oils which is proposed for publication as information. A report of Section D, containing the results of cooperative tests conducted in the development of this method is appended to this report.

Research Division VIII on Volatility (G. G. Lamb, chairman) incorporates the work previously handled by Subcommittees VIII, XII, and XXI, Section II of Technical Committee A, and Section IV of Technical Committee J.

Section B on Flash Point (E. W. Dean, chairman) is planning an investigation of the Tag open cup method of test, with the possibility of its standardization.

Section E on Vacuum Distillation (J. F. Middleton, chairman) developed the Proposed Method of Test for Reduced Pressure Distillation of Petroleum Products and has proposed its publication as information. This method is the result of several years' active cooperative work in a difficult field. Further revision of this method will undoubtedly be necessary before it is ready for acceptance as tentative. Work is simultaneously under way toward the development of a vapor temperature-pressure correlation which can ultimately be used for the conversion of distillation curves obtained at 10 mm. or less to atmospheric pressure.

Research Division IX on Color (formerly Subcommittee VI) (H. M. Hancock, chairman) is recommending the continuation as tentative of the Methods of Test for Color of U. S. Army Motor Fuel (All-Purpose) by Means of an A.S.T.M. Color Standard (D 976 - 48 T) and for Color of Lubricating Oils and Petroleum by means of the A.S.T.M. Union Colorimeter (D 155 - 45 T). Work is progressing on a project for establishing fundamental specifications for the A.S.

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[10] Issued as a separate publication *STP No. 111*, Jan.-Feb., 1951.

T.M. union colorimeter standards. This division is also contemplating revision of the Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (D 156 – 49) to include pharmaceutical white oils and petrolatums.

Research Division X on Corrosion Tests (F. D. Tuemmler, chairman) is continuing to expand and improve the Method of Test for Free and Corrosive Sulfur in Petroleum Products (D 130 – 49 T). A further revision of this test, which changes the method of describing the appearance of the copper test-strip and includes a specific A.S.T.M. copper strip corrosion scale has been prepared and is being proposed by the committee. As a result of the general acceptance of Method D 130 – 49 T by Technical Committees A, E, and J, Research Division X recommends, this year, the withdrawal of the old version of this method, Standard D 130 – 30.

A new Section B on Humidity Cabinet Corrosion Test, of Research Division X, has been established.

Subcommittee I on Pharmaceutical Tests (C. F. W. Gebelein, chairman) is currently working on a means of improving the Method of Test for Penetration of Petrolatum (D 937 – 49 T). The possibility of employing a stable standard reference material for penetrometer calibration is being investigated. Subcommittee I is also carrying out additional work on the Method of Test for Carbonizable Substances in White Mineral Oil (D 565 – 45).

Subcommittee III on Paraffin Wax (J. B. Rather, Jr., chairman) is currently working on methods of test for microcrystalline wax. It is determining whether or not the Method of Test for Oil Content of Paraffin Wax (D 721 – 49 T) can be modified so as to be suitable for the handling of these waxes. The future activities of this subcommittee are being re-evaluated as a result of the organization of Technical Committee M on Petroleum Wax.

Subcommittee XVII on Plant Spray Oil Tests (L. Mittelman, chairman) is working on refinements in the Method of Test for Unsulfonated Residue of Plant Spray Oils (D 483 – 40), and for Distillation of Plant Spray Oils (D 447 – 41), which will include recommendations received from the Pacific Insecticide Institute.

Subcommittee XXIV on Petroleum Sulfonates (C. F. W. Gebelein, chairman) is recommending that the Methods of Analysis of Petroleum Sulfonates (D 855 – 46 T) be continued as tentative. This subcommittee is currently working on a revised method for determining sulfonate molecular weight and is writing up several improvements in testing procedure for use in a projected revision of the method.

Special Subcommittee on Extreme-Pressure Properties Measurement (Harry Levin, chairman) is continuing its detailed cooperative study of the technique of operating the Timken testing machine for the purpose of writing a procedure that will give an adequate degree of reproducibility.

Coordinating Division on Nomenclature (S. S. Kurtz, Jr., chairman) is actively engaged in obtaining industry-wide recognition of the definitions which have been published as A.S.T.M. Standard D 288 – 49, Definitions of Terms Relating to Petroleum.

Coordinating Division on Test Methods (F. D. Tuemmler, chairman) is currently preparing a draft of a recommended practice on the use of precision data in D-2 methods. This is based on a similar publication by the Institute of Petroleum. This recommended practice will embody precision concepts for the terms repeatability and reproducibility.

Coordinating Division on Significance

of Tests (F. D. Tuemmler, chairman) is making considerable headway in its major revision of the A.S.T.M. publication "Significance of Tests of Petroleum Products." The division is receiving excellent cooperation from the officers of the various technical committees.

Coordinating Division on Research (H. P. Ferguson, chairman) held its organization meeting in February, 1950. It has prepared a letter for transmittal to all committee chairmen, outlining the scope of the division and requesting their cooperation in establishing and continuing its activities.

RECOMMENDATIONS AFFECTING STANDARDS AND TENTATIVES

The proposals of the technical committees, divisions and subcommittees, as outlined above, have been presented for discussion in open meetings of Committee D-2 and have been submitted subsequently to letter ballot of the committee. The results of this ballot will be available at the Annual Meeting and made a matter of record at A.S.T.M. headquarters. In no case did a proposal receive a negative vote of 10 per cent or more. On the basis of these results, Committee D-2 makes the recommendations to the Society as set forth in the following sections.

Committee D-2 further proposes that those recommendations concerning the adoption of tentatives as standards and the revision of standards for immediate adoption, which are approved by the Society, be referred to ASA Sectional Committee Z11 on Petroleum Products and Lubricants for approval as American Standards.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

Committee D-2 recommends that the following proposed methods of test be published as information, as appended hereto.[11]

Appendix I. Test for Hydrocarbon Type Analysis of Diesel Fuels by Silica Gel Adsorption.
Appendix II. Test for Bromine Number of Petroleum Distillates (Color Indicator Method).
Appendix III. Test for Bromine Number of Petroleum Distillates (Electrometric Method).
Appendix IV. Test for Measurement of Refractive Index and Refractive Dispersion of Hydrocarbon Liquids.
Appendix V. Test for Measurement of the Density of Knock Test Standard Normal Heptane and *Iso*octane by the Pycnometer.
Appendix VI. Test for Pour Stability Characteristics of Winter Grade Motor Oils.
Appendix VII. Test for Reduced Pressure Distillation of Petroleum Products.
Appendix VIII. Definition and Specifications for Tractor Fuels.

NEW TENTATIVES

Committee D-2 recommends that the following nine methods be accepted for publication as tentative:

Tentative Methods of Test for:[12]

Apparent Viscosity of Lubricating Greases (see Appendix II, 1949 Annual Report of Committee D-2),[12a]
Boiling Point Range of Polymerization Grade Butadiene (see Appendix VII, 1949 Report of Committee D-2),[6]
Carbonyl Content of Butadiene,
Factors and Tables for Volume Correction and Specific Gravity Conversion of Liquefied Petroleum Gases (see Appendix V, 1948 Report of Committee D-2,[7a] except that values above 0.600 will be deleted and values for 0.590 and 0.595 revised based on current information and calculations.
Phosphorus in Lubricating Oils, Lubricating Oil Additives, and Their Concentrates (see Appendix VIII, 1949 Report of Committee D-2),[9a]
Analysis of 60 Octane Number *Iso*octane-Normal Heptane A.S.T.M. Knock-Test Reference Fuel Blends by Infrared Spectrophotometry,
1,3-Butadiene in C_4 Hydrocarbon Mixtures by Ultraviolet Spectrophotometry,
Acidity of Residue from Distillation of Gasoline and of Petroleum Solvents, and
Water Tolerance of Aircraft Fuels.

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[11] See pp. 264 to 311.

[12] These methods were accepted by the Society as tentative and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[12a] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 338 to 344 (1949).

REVISION OF TENTATIVES

Committee D-2 recommends revision of the following eleven tentatives as indicated:

Tentative Specifications for:

D 439 - 49 T.[13] Gasoline, revised as follows:

Table I.—Under the heading "Vapor Pressure, max. psi. at 100 F.", add a footnote *e* applicable to the 13.5 values in the column headed "W", to read: "[e]The vapor pressure shall be 15.0 psi. in Sections 1 and 2 except in March and November in Section 2."

Also under the heading "Vapor Pressure, max. psi. at 100 F." add an additional Footnote *f* applicable to all vapor pressures for all types of gasoline listed in Table I to read:

"[f] Lower maximum vapor pressures may be required for operations at high altitudes or where abnormally high fuel system temperatures are encountered as in some heavy-duty equipment (see Section 1 (*b*)) and in some heavy-duty operations."

Continue the present motor method octane number minimum limits unchanged and delete the date (1940) shown under footnote *b*.

D 975 - 48 T.[13] Diesel Fuel Oils, revised as appended hereto.[14]

D 910 - 48 T.[13] Aviation Gasolines.

Table I. In part 2, line 4, corrosion (copper strip), change the second column to read "No worse than No. 1 comparison standard;" also delete the line in the third column reading "Section 9(*l*)." Also, in footnote *a*, delete the word "However" in the second sentence.

Change footnote *c* to read "The only blue dyes which shall be present in the finished fuel shall consist essentially of alkylamino or arylamino derivatives of anthraquinone."

Change footnote *f*, sentence 2, to read "The balance shall contain no added ingredients other than kerosine and blue dye, which shall consist essentially of alkylamino or arylamino derivatives of anthraquinone."

Section 9 (*e*), Net Heat of Combustion.—Delete the text of this section and substitute a reference to the Standard Method of Test for Thermal Value of Fuel Oil (A.S.T.M. Designation: D 240).[13]

Section 9 (*g*), Acidity of Distillation Residue.—Delete the text of this section and substitute a reference to the Tentative Method of Test for Acidity of Residue from Distillation of Gasoline and of Petroleum Solvents (A.S.T.M. Designation: D 1093 - 50 T).[15]

Section 9 (*i*), Corrosion (Copper Strip).—Delete the text of this section and substitute a reference to the Tentative Method of Test for Free and Corrosive Sulfur in Petroleum Products, Copper Strip Test (A.S.T.M. Designation: D 130 - 50 T).

Section 9 (*l*), Visible Lead Precipitate.—Delete this section in its entirety, and reletter the subsequent paragraphs of Section 9.

Section 9 (*o*), Water Tolerance.—Delete the text of this section and footnote 12, and substitute a reference to the Tentative Method of Test for Water Tolerance of Aircraft Fuels (A.S.T.M. Designation: D 1094 - 50 T).[15]

Tentative Methods of:

D 665 - 49 T.[13] Test for Rust-Preventing Characteristics of Steam Turbine Oil in the Presence of Water, revised as appended hereto.[14]

D 90 - 47 T.[13] Test for Sulfur in Petroleum Products by the Lamp-Gravimetric Method. Change Section 8 (*a*) to read as follows:

"8. (*a*) Transfer the absorber liquid to a 400-ml. beaker. Rinse the absorber thoroughly with distilled water and add the rinsings to the beaker. Filter the solution to remove any foreign material, receiving the filtrate in a 400-ml. beaker having a mark to indicate 75 ml. Add 2 ml. of HCl, heat to boiling, and add 10 ml. of $BaCl_2$ solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 min. thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 ml. as indicated by a mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow to cool 1 hr. before filtering."

D 808 - 49 T.[13] Test for Chlorine in Lubricating Oils by Bomb Method. Change title to read: "Test for Chlorine in Lubricating Oils and Greases by the Bomb Method." Change Section 1 to read as follows:

"1. This method describes the procedure for the determination of chlorine in lubricating oils and greases, including new and used oil and greases containing additives and additive concentrates. Its range of applicability is 0.1 to 50 per cent chlorine for oils and 0.1 to 10 per cent chlorine for greases."

Change Section 4 (*c*) under Reagents to read: "Silver Nitrate Solution (50 g. per liter).—Dissolve 50 g. of $AgNO_3$ in distilled water and dilute to 1 liter."

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[13] 1949 **Book of A.S.T.M. Standards, Part 5.**

[14] This revised method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 5.

[15] This method was accepted by the Society as tentative and appears in the 1950 Supplement to Book of ASTM Standards, Part 5.

Change Section 5 (*e*) under procedure to read: "(*e*) Determination of Chlorine.—Filter the solution through a qualitative paper (if the solution is cloudy, the presence of lead chloride is indicated and the solution should be brought to a boil before filtering), and collect in a second 600-ml. beaker. Add HNO_3 (1:1) drop by drop until acid to litmus. Heat the solution to about 140 F. (60 C.), and while protecting the solution from strong light, add gradually with stirring a solution of silver nitrate (50 g. per liter) until precipitation is complete. Heat to incipient boiling and retain at this temperature until the supernatant liquid becomes clear. Test to insure complete precipitation by adding a few drops of the silver nitrate solution. If more precipitation takes place, repeat the above operation. Allow the beaker and contents to stand in a dark place for at least an hour. Filter the precipitate ....."

D 809 - 44 T.[13] Chemical Analysis for Phosphorus in Lubricating Oils. Include the following note between title and scope: "The committee responsible for the development of this method intends to replace it by the more generally applicable 'Tentative Method of Test for Phosphorus in Lubricating Oils, Lubricating Oil Additives, and Their Concentrates' (A.S.T.M. Designation: D 1091 - 50 T)."[15]

D 1016 - 49 T.[13] Test for Determination of Purity from Freezing Points. Add new Section 8, *n*-Hexane, as follows:

"8. (*a*) The freezing point is determined from freezing curves with the cage stirrer, with a cooling bath of liquid nitrogen (or liquid air) with a cooling rate of 0.3 to 0.8 C. per min. for the liquid near the freezing point, and with crystallization induced, immediately below the freezing point, by means of a cold rod.

"(*b*) A sample of 50 ml. (measured at room temperature) is obtained directly from its original container by means of a pipet or by pouring into a graduated cylinder."

"(*c*) for *n*-hexane, the freezing point for zero impurity, in air at one atmosphere, is:

$$t_{f_0} = -95.347 \pm 0.010 \text{ C.}$$

and the cryoscopic constants are:

A = 0.04975 mole fraction per degree.

B = 0.0038 mole fraction per degree."

"(*d*) The cryoscopic constants given in Paragraph (*c*) are applicable to samples of *n*-hexane having a purity of about 95 mole per cent or better, with the usual impurities and with no one impurity present in an amount which exceeds the composition of its eutectic with the major component.

"(*e*) The estimated uncertainty in the calculated value of the purity is as follows, in mole per cent:

Calculated Purity, mole per cent	Uncertainty, plus or minus, mole per cent
over 99.5	0.05
99.0 to 99.5	0.06
98 to 99	0.07
97 to 98	0.08
96 to 97	0.10
95 to 96	0.12"

Renumber Sections 8 to 19, inclusive, as Sections 9 to 20.

In the new Section 9 on *n*-heptane, Paragraph (*c*), change the equation to read: "$t_{f_0} = -90.610 \pm 0.010$ C."

D 893 - 48 T.[13] Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils. Change Section 1 on scope to read as follows:

"1. This method of test is intended for the determination of normal pentane and benzene insoluble matter in used lubricating oil."

D 130 - 49 T.[13] Test for Free and Corrosive Sulfur in Petroleum Products. Change this method as follows:

In Section 1 on Scope add the word "certain" just before the words "other petroleum products."

Delete in its entirety the Appendix on Interpretation of Results.

Section 7, revise to read as follows: " 7. *Interpretation of Results*—Report the sample as passing the test if the appearance of the test strip matches that of the comparison standard prescribed by the specifications or is similar to the appearance of one of the lower numbered comparison standards. The numerical and descriptive designations of the recommended comparison standards are given in Table I." (The accompanying Table II.)

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-2 recommends that the following fifteen tentatives which have been published for two years or longer without revision be continued in their present status:

Tentative Specifications for:

D 396 - 48 T. Fuel Oils,

Tentative Methods of:

D 874 - 47 T. Test for Sulfated Residue from New Lubricating Oils,

D 892 - 46 T. Test for Foaming Characteristics of Crankcase Oils,

D 943 - 47 T. Test for Oxidation Characteristics of Inhibited Steam Turbine Oils,

D 972 - 48 T. Test for Evaporation Loss of Lubricating Greases and Oils, D 613 - 48 T. Test for Ignition Quality of Diesel Fuels by the Cetane Method,

D 526 - 48 T. Test for Tetraethyl Lead in Gasoline,

TABLE II.—A.S.T.M. COPPER STRIP CORROSION SCALE.

Number	Designation	Description of Strip Appearance
0	No tarnish	Almost the same as a freshly polished strip.
1	Slight tarnish[a]	Orange
2	Moderate tarnish	Any solid color or combination of colors,[b] except those listed elsewhere in this scale.
3	Dark tarnish	Peacock (multi-colored with green but no gray).
4	Slight corrosion	Peacock (multi-colored with green and gray).
Greater than 4	Pronounced corrosion	Solid gray, brown, or black with or without peeling.

[a] Sometimes referred to as "no more than extremely slight corrosion."

[b] Solid colors normally range through red, lavender, silver, brassy and sometimes blue in increasing order of thickness of the film deposited on the strip.

D 875 - 46 T. Test for Olefins and Aromatics in Petroleum Distillates,

D 611 - 47 T. Test for Aniline Points and Mixed Aniline Points of Petroleum Products,

D 94 - 48 T. Test for Saponification Number of Petroleum Products by Color-Indicator Titration,

D 974 - 48 T. Test for Neutralization Value (Acid and Base Numbers) by Color-Indicator Titration,

D 445 - 46 T. Test for Kinematic Viscosity,

D 976 - 48 T. Test for Color of U. S. Army Motor Fuel (All-Purpose) by Means of an A.S.T.M. Color Standard,

D 155 - 45 T. Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter, and

D 855 - 46 T. Analysis of Petroleum Sulfonates.

ADOPTION OF TENTATIVES AS STANDARD

Committee D-2 recommends that the following four tentatives be approved for reference to letter ballot of the Society for adoption as standard, four without revision and one with revision as indicated:

Tentative Methods of:

D 971 - 48 T. Test for Interfacial Tension of Oil Against Water by the Ring Method,

D 942 - 47 T. Test for Oxygen Stability of Lubricating Greases by the Oxygen Bomb Method,

D 973 - 48 T. Test for Determination of Butadiene Content of Polymerization-Grade Butadiene, and

D 939 - 47 T. Test for Saponification Number of Petroleum Products by Potentiometric Titration, with the following revisions:

Section 8 (*c*).—Change to read as follows:

"(*c*) Disconnect the condenser and wash the exposed condenser and beaker surfaces with 10 to 20 ml. of titration solvent."

Section 8 (*d*).—Change to read as follows:

"(*d*) While the contents of the saponification beaker are still hot, quickly transfer the liquid phase by decantation to a second 250-ml., tall-form, lipless beaker, rinsing the saponification beaker and insoluble residue with three 5-ml. portions of titration solvent; add the rinsings to the second beaker, cover with a watch glass, and cool the beaker and contents to room temperature. Add 100 ml. of carbon dioxide-free distilled water to the original saponification beaker without delay after separation and heat just to boiling. Cover the beaker with a watch glass and cool to room temperature. Titrate the solutions as described in Paragraphs (*e*) and (*f*) within 2 hr. after the end of the saponification period."

Section 8 (*b*).—Change the second sentence to read as follows:

"Without transfer of the saponification solution, cover the beaker with a watch glass, cool to room temperature, and titrate with 0.2 *N* alcoholic HCl directly in the saponification beaker within 2 hr. after the end of the saponification period."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

Committee D-2 recommends for immediate adoption revisions in the following three standards, and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Standard Methods of:

D 240 - 39.[13] Test for Thermal Value of Fuel Oil, revised as follows:

Title.—Change to read "Test for Heat of Combustion of Liquids by Bomb Calorimeter."

Section 1.—Change to read: 1. *Scope.*—This method describes procedures for the determination of heat of combustion of aviation gasoline and the thermal value of fuel oils by means of a bomb calorimeter."

Section 3 (b).—Add the following note: "*Note 1*.—Acceptable procedures for handling volatile liquids include those described in the following reports: Richards and Barry, *Journal*, Am. Chemical Soc., **37**, 993 (1915); Jessup, *Journal of Research*, Nat. Bureau Standards, **18**, 115 (1937); Proson and Rossini, *Journal of Research*, Nat. Bureau Standards, **27**, 289 (1941); Dean, Williams and Fisher, *Industrial and Engineering Chemistry*, Analytical Edition, **16**, 182 (1944); LeTourneau and Matteson, *Analytical Chemistry*, **20**, 663 (1948)."

Section 6.—Change to read as follows: "6. (*a*) Calculate the gross heat of combustion of the sample by means of the following equation:

Gross Heat of Combustion, Btu. per lb. =

$$\frac{1.8\,(tw - e_1 - e_2 - e_3)}{g} + 1.6\,(t_e - 25)$$

where:

t = corrected temperature rise, degrees Centigrade, as calculated in Section 5,

t_e = maximum temperature, degrees Centigrade, reached after firing corrected for thermometer errors,

w = water equivalent of calorimeter,

e_1 = corrections, calories, of nitric acid formed (230 cal. per g.),

e_2 = corrections, calories, of sulfur content (1300 cal. per g.) for differences in heats of formation of sulfur dioxide and aqueous sulfuric acid (see Note 2),

e_3 = corrections, calories, for iron firing wire (1600 cal. per g.), and

g = weight of sample in grams.

NOTE 2.—This represents an additional correction as sulfuric acid has been titrated and calculated as nitric acid.

(*b*) Report as the thermal value of fuel oil, the gross heat of combustion as calculated in Paragraph (*a*).

(*c*) Report the net heat of combustion of aviation gasoline, calculated from the gross heat of combustion by means of the following equation:

Net Heat of Combustion, Btu. per lb. = 4310 + (0.7195 × gross heat of combustion)."

Section 7.—Change to read as follows: "7. *Precision*.—Results should not differ from the mean by more than the following amounts:

	Repeatability (Same Operator and Apparatus), per cent of mean	Reproducibility (Different Operator and Apparatus), per cent of mean
Gross or net Heat of Combustion, Btu. per lb.. .	0.3	0.5

D 129 – 49.[13] Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method, revised as follows:

Section 1.—Change to read: "1. This method describes the procedure for the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases, that cannot be burned completely in a wick lamp."

Section 5(d).—Revise the first sentence to read "Rinse the interior of the bomb, the oil cup, and the inner surface of the bomb cover with a fine jet of distilled water, and collect the washings in a 600-ml. beaker having a mark to indicate 75 ml."

Section 5(e).—Revise the first four sentences to read: "To the combined washings add 10 ml. of saturated bromine water and evaporate to approximately 200 ml. on a hot plate or other source of heat. Adjust the heat to maintain slow boiling of the solution and add 10 ml. of $BaCl_2$ solution, either in a fine stream or dropwise. Stir the solution during the addition and for 2 min. thereafter. Cover the beaker with a fluted watch glass and continue boiling slowly until the solution has evaporated to a volume of approximately 75 ml. as indicated by a mark on the beaker. Remove the beaker from the hot plate (or other source of heat) and allow to cool for 1 hr. before filtering."

Section 7.—In the table add a footnote reference[a] referring to ranges of 1.0 to 2.0 and 2.0 to 3.0, as follows:

"[a] For lubricating greases in the range of 1.0 to 3.0 per cent sulfur the repeatability and reproducibility is 0.05 and 0.10, respectively."

D 381 – 49.[13] Test for Existent Gum in Gasoline, revised as appended hereto.[14]

WITHDRAWAL OF STANDARD

Committee D-2 recommends the withdrawal of Standard Method D 130 – 30, Test for Detection of Free Sulfur and Corrosive Sulfur Compounds in Gasoline.[13]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, O. L. Maag.
First Vice-Chairman, L. C. Beard, Jr.
Second Vice-Chairman, H. M. Smith.
Secretary, W. T. Gunn.

This report has been submitted to letter ballot of the committee, which consists of 95 voting members; 72 members returned their ballots, of whom 67 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

O. L. MAAG,
Acting Chairman.

W. T. GUNN,
Secretary.

RESULTS OF TESTS ON PROPOSED METHOD FOR DETERMINING POUR STABILITY CHARACTERISTICS OF WINTER GRADE MOTOR OILS

Submitted by Section D of Research Division VII

Section D of Research Division VII on Cloud and Pour Test (formerly Subcommittee XVI) recommends that the appended Proposed Method of Test for Determining Pour Stability Characteristics of Winter Grade Motor Oils be published as information only.

The inadequacy of the A.S.T.M. Method of Test for Cloud and Pour Points (D 97 – 39)[1] in predicting the pour point stability of winter grade lubricating oils containing pour point depressants has long been recognized. Accordingly, the members of this section have been concerned for a number of years with the development of a laboratory test capable of more accurately predicting the pour point stability of such oils. The first method developed and published as information only was a Proposed Method of Estimating Maximum Pour Points of Lubricating Oils Containing Pour Point Depressants.[2] Field data on the 19 reference oils under study were too limited at that time to permit a suitable correlation with laboratory test results. Cooperative field testing by three laboratories was continued, and a complete summary of field and laboratory tests was published in 1946.[3]

The conclusions from the work to that point were:

1. The proposed method had the advantage of utilizing present A.S.T.M. pour test equipment and of being performed in 6 to 8 hr.

2. Based on field test data, it represented a definite improvement over the present A.S.T.M. Method D 97 as a means of predicting pour point stability. Correlation, based on approximately 10 F. agreement between field and laboratory results, showed 63 per cent agreement with the proposed test and only 10 per cent with A.S.T.M. Method D 97.

3. The method appeared to be quite consistent in giving warning in the case of very unstable oils.

4. The proposed method had a definite limitation in that it failed to indicate the relative performance of a series of oils when maximum pour points were compared with frequency of solidification in the field. For example, one oil may have been solid once or twice in the field while another oil may have been solid 300 times, yet the test tended to rate them equally poor. In other words, too many oils which normally show good field performance failed in the test. This has been observed repeatedly by a number of the section members who have conducted extensive tests on other than the reference oils.

In addition to the published conclusions, it has been observed that increasingly lower pour points can be obtained in the proposed maximum pour point test by increasing the dosage of pour point depressant above that required for a specified A.S.T.M. pour point. However, these ***improved laboratory blends*** may

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[1] The present method D 97 – 47 appears in the 1949 Book of A.S.T.M. Standards, Part 5, p. 736; also in "ASTM Standards on Petroleum Products and Lubricants, p. 50.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 244 (1945).

[3] J. J. Giammaria, "Laboratory Determined Pour Points of Lubricating Oils as Related to Ability to Flow Under Field Storage Conditions," ASTM Bulletin, No. 138, January, 1946, p. 44.

TABLE I.—SUMMARY OF FIELD AND LABORATORY TESTS

All results are in degrees Fahrenheit

	OIL SAMPLE																						
	HC-1	12	46-285	15	8	11	14	9	18	10	PL-246	HC-2	7	17	46-284	13	HC-3	IC-4	LC-6	16	HC-12	PL-245	IC-5
A.S.T.M. cloud point	+32	+28	+32	+32	+44	+22	+24	+22	+30	+22	+22	+32	+14	+20	+32	+18	+32	+18	+8	+16	+32	+28	+12
A.S.T.M pour point	−25	−25	+10	−20	−30	−10	−30	−10	−25	−10	−30	−15	−25	−25	−30	−25	−20	−20	−20	−25	+15	<−30	−30
Maximum pour point..	+15	+15	+5	+15	0 or above	+15	−20	+15	+5	+20	−10	+10	−25	+10	0	−15	+15	0	−10	−20	+25	−30	−15
Stable pour point,																							
Cycle A:																							
Laboratory A	+25	+15	+25	+25	+15	+5	+5	−10	+5	−15	<−20	<−20	−15	<−20	<−20	<−20	<−20	<−20	<−20	−15	<−20	<−20	<−20
Laboratory D.....	+25	+20	+15	+25	+25	+10	−15	−15	+10	−15	<−20	<−20	...	<−20	<−20	<−20	<−20	−15	<−20	<−20	<−20	<−20	<−20
Cycle C:																							
Laboratory A.....	+25	+10	+25	+10	<−20	+15	−5	+5	<−20	0	−20	−15	−15	+10	+5	0	<−20	0	<−20	−5	<−20	<−20	<−20
Laboratory D.....	+25	+10	+25	+15	<−20	+10	+10	−5	<−20	0	−15	−15	...	+10	<−20	−5	<−20	+10	<−20	−15	<−20	<−20	<−20
Field Tests[a]																							
Highest Solid Point	+26	+26	+26	+23	+28	+20	+13	+20	+12	+9	+12	+16	+5	+14	+5	−1	+20	−2	+5	−8	−12	−29	<−19
Total Times Solid...	1315	743	680	606	590	193	113	95	52	45	25	15	13	10	9	8	5	3	2	2	2	1	0

[a] 1943–1948 inclusive.

TABLE II.—RESULTS OF CLOUD AND POUR POINT TESTS.
All results are in degrees Fahrenheit

Cooperating Laboratories	Oil Sample															
	1449-4-3	1446-160-2	1446-160-3	1449-6-14	5W	10W(a)	10W(b)	20W	A-2124	A-2123	A-2125	A-2122	RN-95-X	XRN-113-A	XRN-114-A	RN-117-B
A.S.T.M. Cloud Points—Method D 97																
A	+6	+32	+16	+16	+22	+10	+20	+22	+20	+18	+24	+38	+8	+20	–	+28
B	+4, +6	+30, +32	+18, +20	+16, +20	+22	+10, +14	+22	+24, +28	+22, +20	+22, +20	+28, +26	+42	+10	+24, +22	+30	+28, +30
C	0	+28	+16	+16	+18	+6	+16	+22	+18	+16	+22	+38	+6	+20	–	+26
D	+6, +8	+30, +32	+18	+14, +12	+22, +20	+12, +10	+18	+24, +20	+18, +16	+16	+20, +22	+40	+10	+20, +22	–	+30, +28
E	0	+32	+18	+18	+22	+6	+18	+26	+20	+18	+24	+38	+8	+22	+32	+26
Average	+4	+30	+18	+16	+22	+10	+18	+24	+20	+18	+24	+40	+8	+22	+32	+28
A.S.T.M. Pour Points—Method D 97																
A	<−35	−25	−25	0	<−35	−15	−5	0	+5	+15	−20	−15	<−35	−15	−30	−20
B	−35	−10	−25	−10	<−35	−25	−20	−10	−10	+15	−20	−15	<−35	−10	−30	−25
C	<−35	−15	−30	−5	<−35	−20	−15	−10	−10	+10	−15	−15	<−35	−10	−30	−20
D	<−35	−15	−35	−5, 0	<−35	−20, −15	−10, −5	−5, 0	−5	+15	−20	−20	<−35	−15	<−30	−25
E	<−30	−20	−30	−5	<−30	−25	−10	−15	−10	+10	−15	−10	<−30	−10	<−30	−25
Average	<−35	−15	−30	−5	<−35	−20	−10	−5	−5	+15	−20	−15	<−35	−10	−30	−25

MAXIMUM POUR POINTS—PROPOSED METHOD[a]																
A	<−35	−15	−10	+15	<−35	0	+15	+20	+5	+20	+15	+10	<−35	+15	−20	+5
B	<−35	+5, 0	−15	+10	<−35	0, −5	+15, +10	+20	+5, 0	+20, +15	+10, +15	+15, +10	<−35	+15, +10	−20	0
C	<−35	+5	−15	+15	−30	0	+15	+20	+10	+15	+15	+15	<−35	+15	−15	+5
D	<−35	+5	−10	+10	<−35	0	+20	+20	+10	+20	+20	+20	<−35	+20	−15	+10
E	<−30	0	−20	−5	<−30	−5	+15	+20	+10	+10	+15	+10	<−30	+15	−25	0
Average	<−35	0	−15	+10	<−35	0	+15	+20	+5	+15	+15	+15	<−35	+15	−20	+5
STABLE POUR POINT, CYCLE A—PROPOSED METHOD[b]																
A	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	+5, +5	<−20, <−20	<−20, <−20	−15, <−20	+25, +20	0, −5
C	<−20	−10	−15	−15	<−20	<−20	<−20	<−20	<−20	<−20	+15	<−20	<−20	<−20	+25	<−20
D	<−20	−15	<−20	<−20	<−20	<−20	<−20	−15	<−20	<−20	−5	<−20	<−20	−15	+25	0
Average[c]	<−20	−15	<−20	<−20	<−20	<−20	<−20	<−20	<−20	<−20	+5	<−20	<−20	<−20	+25	−5
STABLE POUR POINT, CYCLE C—PROPOSED METHOD[b]																
A	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	−15, 0	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	<−20, <−20	−20, <−20
C	<−20	<−20	−15	−15	<−20	<−20	<−20	−15	<−20	<−20	−10	<−20	<−20	−15	<−20	−10
D	<−20	−15	<−20	−15	<−20	<−20	<−20	<−20	<−20	<−20	−15	<−20	<−20	−15	<−20	−15
Average	<−20	<−20	<−20	<−20	<−20	<−20	−15	<−20	<−20	<−20	−15	<−20	<−20	<−20	<−20	−15

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[a] As published for information in Appendix I to the 1945 Report of Committee D-2.

[b] As published for information in Appendix VI to this report, see p. 696.

[c] Where necessary for computing average, a value of −25 was used for <−20.

give poor pour stability performance in the field. In other words, with certain pour point depressants, the use of the proposed test as a guide for required concentration for desired pour stability may furnish misleading information and result in a compounded oil which is inferior in pour stability in the field to one containing a smaller concentration of pour depressant.

In view of the limitations of the proposed Maximum Pour Point Method, work has continued on an improved method based on slower cooling and warming rates during temperature cycling. A method which showed promise was first presented at the S.A.E. National Fuels and Lubricants Meeting on November 7, 1946, and was published in 1947.[4] This proposed method has been under study by the members of this section during the past several years and the results of these studies are published at this time for information.

The first cooperative work on the method was carried out by two laboratories having suitable equipment. The method was applied to the 19 original reference oils and four additional oils which were submitted in 1946. The available results on these 23 oils by the latest proposed method are shown in Table I, together with A.S.T.M. cloud points, A.S.T.M. pour points, maximum pour points, and complete field tests. The oils have been arranged in order of their field performance based on the frequency of solidification over a 5-yr. field testing period. The frequency of solidification is important in classifying the oils. Thus, oils HC-1 to PL-246 may be considered as definitely unstable oils, having shown solid points at most of the stations at some time. However, oils HC-2 to IC-5 are oils of good stability, considering the length of the field tests and the low frequency of solidification. For example, reviewing the field history of oil HC-2, it was found that the solid point shown occurred only at two stations during the winter of 1943–1944, the oil being fluid at all other stations at all times for the remainder of the test. Similarly, oil HC-3 was reported solid at +20 F. only twice at one station during the entire field test. If a correlation between field and laboratory results is based strictly on the highest field solid points, it is seen that both the previous and currently proposed tests are in fairly good agreement with the field tests. Based on agreement within approximately 10 F., the maximum pour points agree with the field test results in 16 of 23 cases (including No. 8). With the currently proposed method, cycle A agrees in 13 of 22 cases, cycle C in 14 of 23 cases. Using the highest stable pour point obtained in either of the two cycles, agreement occurs in 17 of 23 cases. Average results for cycle A or cycle C were not used because of the limited number of tests. Average results for combined cycle A and cycle C tests were not used because of the nature of the tests, the cycle A test being particularly applicable for high cloud point oils (+24 F. to +40 F.) and the cycle C test for low cloud point oils (+24 F. and lower).

Referring again to the frequency of solidification as a means of classifying the test oils into two classes, stable and unstable, it is seen that a combination of the two cycles gives a more practical prediction of field performance. With oils HC-1 to PL-246, which may be considered very unstable oils, both the maximum pour test method and the current proposed method generally predict this instability. In the group HC-2 to IC-5, which are considered oils of good stability, the maximum pour test method fails to indicate the relatively good pour stability of oils HC-2, 17, HC-3, HC-12, and possibly IC-5. Thus, both methods appear to be equally accurate in predicting the behavior of very unstable oils, but the currently proposed method appears to give a fairer indication of the field performance to be expected of oils of good pour stability.

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[4] J. G. McNab, D. T. Rogers, A. E. Michaels, and C. E. Hodges, "The Pour Stability Characteristics of Winter Grade Motor Oils," *S A E Quarterly Transactions*, Vol. 2, No. 1, January, 1948, p. 34; C. E. Hodges and D. T. Rogers, "Some New Aspects of Pour Depressant Treated Oils." *The Oil and Gas Journal*, Vol. 46, October 4, 1947, p. 89.

During the past year, a new series of 16 test oils was distributed to seven cooperating laboratories for further testing. A.S.T.M. cloud points, A.S.T.M. pour points, and maximum pour points as reported by five of the laboratories, and cycle A and cycle C results by the appended method reported from three laboratories, are shown in Table II. Average pour tests by the various methods are summarized in Table III. Field tests on the new series of oils are not yet available. It is obvious from the data in Table III that the maximum pour test method is much more severe than the currently proposed method. Results of current field tests on the new series of oils must be awaited to establish further the relative merits of the two proposed tests.

TABLE III.—AVERAGE POUR TESTS BY VARIOUS METHODS.
(From Table II)

Oil Sample	1449-4-3	1446-160-2	1446-160-3	1449-6-14	5W	10W (a)	10W (b)	20W	A-2124	A-2123	A-2125	A-2122	RN-95X	XRN-113A	XRN-114A	RN-117B
Average A.S.T.M. Pour Points...	<−35	−15	−30	−5	<−35	−20	−10	−5	−5	+15	−20	−15	<−35	−10	−30	..
Average Maximum Pour Points...	<−35	0	−15	+10	<−35	0	+15	+20	+5	+15	+15	+15	<−35	+15	−20	+5
Average Stable Pour Points...																
Cycle A....	<−20	−15	<−20	<−20	<−20	<−20	<−20	<−20	<−20	<−20	+5	<−20	<−20	<−20	+25	−5
Cycle C....	<−20	<−20	<−20	<−20	<−20	<−20	−15	<−20	<−20	<−20	−15	<−20	<−20	<−20	<−20	−15

Although the number of tests run on each sample by different laboratories by the latest proposed method is limited, the data indicate that the degree of reproducibility is comparable to that of the maximum pour point method The results in Table I indicate reproducibility between laboratories within 10 F. for either cycle A or cycle C, with the exception of results on oils 14 and 46-284. This is also indicated, with a few exceptions, by the data in Table II. The repeatability of the test based on duplicate runs in laboratory A only was within 5 F. in all but one case, as shown in Table II.

The general opinion of the section members relative to the merits of the appended method is that the test is more useful than either the A.S.T.M. Method D 97 or the previously proposed Maximum Pour Point Method as a means of classifying a given oil relative to pour point stability. The test is admittedly on the mild side, which may result in its occasionally passing an oil of borderline stability. However, this is offset by its more accurate prediction of definitely stable oils. The term prediction should probably be interpreted as meaning an indication of a tendency to revert rather than an actual pour point value.

The pour reversion work of the section members with both the reference oils and a wide variety of other oils has emphasized the inadequacy of A.S.T.M. Method D 97 for predicting the pour points of oils, containing pour point depressants, under conditions of field storage. While Method D 97 satisfactorily predicts the storage characteristics of oils *not* containing pour point depressants, it can be misleading in the case of oils containing pour point depressants due to the phenomenon of reversion. It is hoped that the publication for information of the attached method will result in its wider use in predicting the performance, under field conditions, of oils containing pour point depressants. It also should be of assistance to lubricating oil manufacturers in evaluating the relative merits of pour point depressants.

Respectfully submitted on behalf of Section D of Division VII,

J. J. GIAMMARIA,
Chairman.

APPENDIX I

PROPOSED METHOD OF TEST FOR HYDROCARBON TYPE ANALYSIS OF DIESEL FUELS BY SILICA GEL ADSORPTION[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination of three hydrocarbon types in Diesel fuels, namely: paraffins plus naphthenes, non-aromatic olefins, and aromatics (Note 1). The method is applicable to hydrocarbon mixtures from petroleum, both straight run and cracked in the boiling range 400 to 700 F. (205 to 371 C.) as determined by A.S.T.M. Method D 158, Test for Distillation of Gas Oil and Similar Distillate Fuel Oils.[3]

NOTE 1.—The term aromatics as used in this method includes both monocyclic and polycyclic aromatic compounds and also sulfur, oxygen, and nitrogen compounds that may be associated with the oil. According to the method described these compounds will remain with the aromatic portion of the oil.

Outline of Method

2. A sample is diluted with an equal volume of pentane, percolated through a fixed quantity of silica gel, and the gel washed with pentane until the refractive index of the percolate approaches closely the refractive index of the pentane. Desorption of the aromatic portion with benzene and methyl alcohol is then accomplished. With the refractive index of pentane as an indication of cut point, two fractions are collected. The first fraction washed through with pentane contains the paraffins, naphthenes, and non-aromatic olefins. The second fraction obtained by desorption with benzene and methyl alcohol contains all the aromatics and aromatic olefins, and most of the non-hydrocarbons. As this procedure is used on fractions boiling above 400 F. (205 C.), it is relatively easy to distill the solvents (pentane, benzene, and methyl alcohol) from the separated fractions using a special stripping apparatus. Quantitative recovery of the separated hydrocarbon types is possible. To obtain the content of non-aromatic olefins, a portion of the paraffin-naphthene fraction is tested according to A.S.T.M. Method D 875, Test for Olefins and Aromatics in Petroleum Distillates.[3] As the aromatics have been removed, the contraction obtained by acid absorption will represent the non-aromatic olefins in the paraffin and naphthene fraction. (Note 2).

NOTE 2.—The bromine number method is not recommended for the determination of olefins because of (*a*) the uncertainties of the bromine number determinations in the higher molecular weight hydrocarbons, and (*b*) the uncertainty of the molecular weight of the olefins present. The latter value is necessary

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants.

[2] Published as information, June, 1950.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

to calculate the volume as percentage of olefins from bromine number.

Apparatus

3. (*a*) *Adsorption Column.*—A column for silica gel consisting of a glass tube 5 cm. in inside diameter by 120 cm. in length with a stopcock at the bottom.

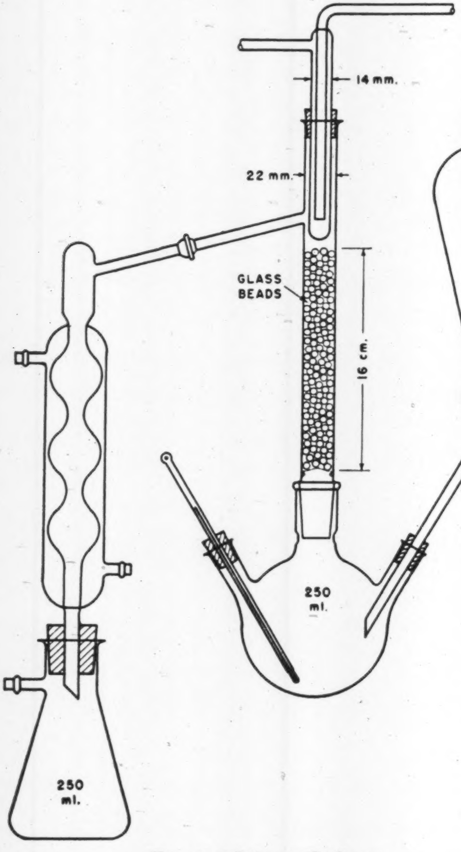

FIG. 1.—Solvent Stripper.

(*b*) *Graduates.*—Several 1-liter graduates.

(*c*) *Stripping Apparatus.*—The stripping apparatus consists of a 250 ml. three-neck round-bottom flask equipped with a 250-ml. separatory funnel in one neck, a short (16 cm.) bead-packed column with cold finger condenser and side arm in the center opening of the flask, and a thermometer in the third opening. The side arm connects to a condenser and thence to a 250-ml. filter flask. A heating mantle is used on the round-bottom flask. A vacuum pump or aspirator is necessary. A suitable assembly is shown in Fig. 1.

(*d*) *Refractometer.*—A refractometer capable of measuring refractive indices reproducible to 0.0002.

(*e*) *Balance.*—A laboratory balance suitable for weighing the samples to plus or minus 0.05 g.

(*f*) *Pycnometer or Westphal Balance.*—The pycnometer described in A.S.T.M.

Method D 941, Measurement of Density of Hydrocarbon Liquids by the Pycnometer,[3] is suitable, or, alternatively, a Westphal balance read to the fourth place may be used.

Reagents

4. (*a*) *Silica Gel.*—Silica gel between 28 to 200 mesh. Its adsorptive capacity should be such that on percolation of 20 ml. of a standard mixture, 20 per cent benzene and 80 per cent methylcyclohexane, through 10 g. of gel in a 20 mm. inside diameter column, no less than 5 ml. of aromatic-free filtrate would be obtained as determined from the refractive index of the first 5-ml. portion of the filtrate.

(*b*) *Solvents.*—Commercial grade normal or isopentane. Benzene and methyl alcohol, such as a c.p. quality and free from high-boiling residual material. Methyl alcohol may require redistillation to remove small quantities of water that interfere in the recovery of the oil.

Procedure

5. (*a*) Tamp a small wad of glass wool into the bottom of the glass column. Weigh out 1 kg. of silica gel and introduce it into the column, tamping the column as it is charged to minimize one cause of channeling of the sample.

(*b*) Weigh 100 ±1 ml. of sample with an accuracy of 0.05 g. and dilute with 100 ml. of pentane.

(*c*) Introduce 500 ml. of pentane into the column and open the bottom stopcock.

(*d*) When the pentane just disappears into the gel, add the diluted sample, with care that none of the oil sample is lost (the sample container should be rinsed with small portions of pentane).

(*e*) As the liquid level falls to the level of the gel, add two 500-ml. portions of pentane, being careful not to disturb the surface of the gel, and then as many 200-ml. portions of pentane as necessary (see Paragraph (*g*)) to displace the adsorbed non-aromatic hydrocarbon portion from the gel, and collect the percolate in a graduated receiver. Regulate the flow at 7 ± 1 ml. take-off per minute.

(*f*) Collect 1-ml. samples of the percolate (for refractive index measurements) after 100 and 1000 ml. of percolate are collected, and at 100-ml. intervals thereafter, until the refractive index is within 0.0002 of the first refractive index measured, that of the pentane itself, which was measured after the first 100 ml. of percolate were collected.

(*g*) Stop the addition of pentane to the top of the column when the refractive index of the percolate is within 0.0002 of the refractive index of the pentane. Then start the desorption of aromatics by adding a mixture of 400 ml. of benzene, plus 100 ml. of methyl alcohol. Follow this with two 500-ml. portions of methyl alcohol to complete the desorption.

(*h*) Two interfaces appear as the adsorption proceeds in the column, the lower interface indicating the position of the benzene front, and the more pronounced upper interface showing the position of the methyl alcohol. When the lower interface is three-fourths of the way down the column, change the receiver, and begin the collection of the second or aromatic fraction. The volume of the first or non-aromatic fraction is usually between 1600 and 2000 ml.

(*i*) The rate of flow of percolate may decrease during the collection of the second fraction, even though the stopcock is fully opened. While the adsorption column is draining, prepare the stripping apparatus for use, and obtain separate tare weights on the 250-ml. round-bottom flask and the column without a condenser.

(*j*) Introduce the percolates containing the non-aromatic oil into the separatory funnel of the stripping apparatus, leaving the stopcock open until the flask is half filled with the solvent-sample mixture. For convenience in handling and to avoid sample losses, a "wash bottle" with rubber pressure bulb is convenient for transferring the sample to the separatory funnel reservoir. Apply heat using a hemispherical mantle and a variable transformer. Adjust the position of the cold finger to give a small amount of reflux (a minimum of about 20 drops per minute) and adjust the heat input to give an initial take-off of from 500 to 1000 ml. per hr. Use a filter flask as a receiver.

(*k*) As the separatory funnel empties, refill with sample until all the percolate has been entered. Wash the transfer container and separatory funnel with a few rinses of pentane. Keep the temperature of the material in the flask constant until boiling almost ceases. At this point, use an empty filter-flask as the solvent receiver and apply vacuum (a water aspirator is a suitable source of vacuum) continuously at the filter-flask connection. After boiling, induced by the pressure reduction, almost ceases, increase the heat until the oil is again brought to a boil or to about 250 F. (121 C.), and then remove the heater from the flask.

(*l*) Weigh the cooled flask and column separately to 0.05 g. Connect the flask to the open column and heat on a water bath at 80 C. for 1 hr. The loss in weight during this operation will ordinarily not exceed 0.2 g. if the stripping was complete. If the loss is high, repeat the heating on the water bath until the loss is less than 0.2 g. If the weight of oil in the column is greater than 0.5 g., pass the stripped sample through the column to obtain a representative sample for physical property determinations. Record the sum weight of the oil in the flask and column as P_w.

(*m*) Treat the fractions from the desorption with benzene and methyl alcohol in a similar manner in the stripping unit. If on stripping the aromatic portion methanol remains on the beads under the reduced pressure, remove it while the oil is actively boiling by applying heat from an infrared lamp to the column until the methanol has evaporated. The last fraction obtained sometimes contains fines from the silica gel, in which case filter this portion into the separatory funnel and then wash filter with pentane. Record the weight of this portion as A_w.

(*n*) Determine the density of the non-aromatic portion, and of the aromatic portion. Record these densities as D_p and D_a, respectively.

(*o*) Test two 10-ml. portions of the non-aromatic portion for olefins according to A.S.T.M. Method D 875. Record the unreacted or residual volume as *R*.

Calculations

6. (*a*) Calculate the volumes of the aromatic and non-aromatic portions as follows:

$$A_v = \frac{A_w}{D_a}$$

$$P_v = \frac{P_w}{D_p}$$

where:

A_v = milliliters of aromatic portion,
A_w = grams of aromatic portion,
D_a = density of aromatic portion,
P_v = milliliters of non-aromatic portion,
P_w = grams of non-aromatic portion, and
D_p = density of non-aromatic portion.

(*b*) Calculate and report the volume per cent of (*1*) aromatics, (*2*) non-aromatic olefins, and (*3*) paraffins plus naphthenes, as follows:

$$A = \frac{100A_v}{P_v + A_v}$$

$$U = \frac{(100 - 10R - C)P_v}{P_v + A_v}$$

$$PN = \frac{100P_v}{P_v + A_v} - U$$

where:

A = volume per cent of aromatics,
U = volume per cent of non-aromatic olefins,
PN = volume per cent of paraffins plus naphthenes,
R = milliliters of unreacted paraffin-naphthene portion (A.S.T.M. Method D 875), and
C = correction factor (A.S.T.M. Method D 875).

Precision

7. Results should not differ from the mean by more than the following amounts:

Hydrocarbon Content, volume per cent	Repeatability One Operator and Apparatus	Reproducibility Different Operators and Apparatus
Paraffins-naphthenes, 65 to 85...	±1.2	±2.0
Aromatics, 35 to 15..	±1.0	±1.4
Non-aromatic olefins, up to 5......	±0.6	±0.9

APPENDIX II

PROPOSED METHOD OF TEST FOR BROMINE NUMBER OF PETROLEUM DISTILLATES (COLOR INDICATOR METHOD)[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St. Philadelphia 3, Pa.

Scope

1. (*a*) This method is intended for the determination of the bromine number of petroleum distillates that are substantially free of material lighter than isobutane and that have 90 per cent distillation points under 620 F. The method is generally applicable to materials that fall within the following limits:

A.S.T.M. 90 per cent Distillation Point, deg. Fahr.	Bromine Number, max.
Under 400	100
400 to 620	40

(*b*) Because of the variable reactivity of olefins, the bromine number obtained on various petroleum distillates is not an absolute measure of the olefins present but rather a criterion of certain properties of the material tested. It is known that high bromine numbers are obtained with polymer gasolines and those containing highly branched olefins or diolefins. Also, samples containing anthracene-type aromatics, sulfur, oxygen, and nitrogen compounds react to a certain extent with bromine under the conditions of the test. Thus, when bromine numbers are converted to olefins, the values obtained may not be definite and may vary from case to case unless the composition of the material tested is well established.

Definition

2. *Bromine Number* is the number of grams of bromine consumed by 100 g. of the sample when reacted under given conditions.

Outline of Method

3. The sample, dissolved in carbon tetrachloride, is treated at room temperature with an excess of bromide - bromate solution in the presence of glacial acetic acid. The excess bromine is reduced with potassium iodide and the liberated iodine determined by titration with sodium thiosulfate solution.

Apparatus

4. *Iodine Number Flasks*, 500-ml., glass-stoppered, will be required.

Reagents

5. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. References to water shall be understood to mean distilled water.

(*a*) *Carbon Tetrachloride.*—Make blank determinations on 5-ml. portions of each lot of carbon tetrachloride as described in Section 7 (*c*).

(*b*) *Acetic Acid, Glacial.*

(*c*) *Bromide - Bromate Solution* (*Standard 0.5 N*).—Dissolve 51.0 g. of KBr and 13.92 g. of $KBrO_3$ (or the equivalent 44.1 g. of NaBr and 12.58 g. of $NaBrO_3$) in water and dilute to 1 liter. To standardize, place 50 ml. of glacial acetic acid and 1 ml. of HCl (sp. gr. 1.18) in a 500-ml. iodine number flask. Chill the solution in an ice bath for approximately 10 min. and, with constant swirling of the flask, add from a 10-ml. calibrated buret 5 ± 0.01 ml. of bromide - bromate solution at the rate of 1 or 2 drops per second. Stopper the flask immediately, shake the contents, place it again in the ice bath, and add 5 ml. of KI solution in the lip of the flask. After 5 min., remove the flask from the ice bath and allow the KI solution to flow into the flask by slowly removing the stopper. Shake vigorously, add 100 ml. of water in such a manner as to rinse the stopper, lip, and walls of the flask, and titrate promptly with $Na_2S_2O_3$ solution. Near the end of the titration, add 1 ml. of starch indicator solution and titrate slowly to disappearance of the blue color. Calculate the normality of the bromide-bromate solution as follows:

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October 1949; Revised, June, 1950.

Normality of bromide - bromate solution

$$= \frac{\text{ml. of } Na_2S_2O_3 \times \text{normality of } Na_2S_2O_3}{5}$$

Repeat the standardization until duplicate determinations do not differ from the mean by more than plus or minus 0.002 *N*.

(*d*) *Potassium Iodide Solution* (*150 g. per l.*).

(*e*) *Sodium Thiosulfate Solution* (*Standard 0.1 N*).—Dissolve 25 g. of $Na_2S_2O_3 \cdot 5H_2O$ in water and add 0.01 g. of Na_2CO_3 to stabilize the solution. Dilute to 1 liter and mix thoroughly by shaking. Standardize by any accepted procedure that determines the normality with an error not greater than plus or minus 0.0002. Restandardize at intervals frequent enough to detect changes of 0.0005 in normality.

(*f*) *Starch Solution.*—Triturate 5 g. of arrowroot starch and 5 to 10 mg. of HgI_2 with 3 to 5 ml. of water. Add the suspension to 2 liters of boiling water and boil for 5 to 10 min. Allow to cool and decant the clear, supernatant liquid into glass-stoppered bottles.

Check Procedure

6. In case of doubt in applying the procedure to actual samples, the reagents and technique should be checked by means of determinations on freshly purified cyclohexene and diisobutene. Make the tests according to the procedure described in Section 7, using as a sample either 0.5 to 1 g. of freshly purified cyclohexene or diisobutene (see Table II) or 8 to 10 g. of 10 per cent by weight solutions of these materials in carbon tetrachloride. If the reagents and technique are correct, values within the following ranges should be obtained:

Standard	Bromine Number
Cyclohexene, purified (Notes 1 and 2).	191 to 199 (Note 3)
Cyclohexene, 10 per cent solution....	19 to 20
Diisobutene, purified (Notes 1 and 2)..	142 to 148 (Note 3)
Diisobutene, 10 per cent solution....	14 to 15

NOTE 1.—Purified samples of cyclohexene and diisobutene may be prepared from Eastman cyclohexene, No. 1043, and Eastman diisobutene, No. P2125, by the following procedure:

Add 65 g. of activated silica gel (28 to 200 mesh)[3] to a column approximately 16 mm. in inside diameter and 760 mm. in length, which has been tapered at the lower end and which contains a small plug of glass wool at the bottom. A 100-ml. buret, or any column that will give a height-to-diameter ratio of the silica gel of at least 30:1, will be suitable. Tap the column during the addition of the gel to permit uniform packing.

To the column add 30 ml. of the olefin to be purified. When the olefin disappears into the gel, fill the column with methanol. Discard the first 15 ml. of percolate and collect the next 10 ml., which is the purified olefin for test of the bromine number procedure. Determine and record the density and refractive index of the purified samples at 20 C. Discard the remaining percolate.

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[3] Silica gel satisfactory for this purpose can be obtained from the Davison Chemical Corp., Baltimore, Md.

Caution.—If distillation of these olefins is employed as a pre-purification step, at least 10 per cent residue should remain in the distillation flask to minimize the hazards from decomposition of any peroxides that may have been present.

NOTE 2.—The reference olefins yielding the above results are characterized by the properties shown in Table I.

NOTE 3.—The theoretical bromine numbers of cyclohexene and diisobutene are 194.8 and 142.3, respectively. Diisobutene typifies a reactive type olefin which undergoes bromine substitution easily and usually gives bromine number values 2 to 6 units higher than theoretical by the check procedure. This condition is recognized with disfavor but considered acceptable for most routine testing of material in this category.

The theoretical value of diisobutene may be more closely approached by rigid control of the time schedule and excess bromine concentration to the minima permitted by the detailed procedure. It is to be expected that the accuracy of the bromine number determination for the reactive type olefins will be improved as the check procedure with diisobutene approaches the theoretical value of 142.3.

TABLE I.—PHYSICAL PROPERTIES OF PURIFIED OLEFINS.

Compound	Boiling Point deg. Cent	Density at 20 C., g. per cu. cm.	Index of Refraction, *D* Line at 20 C.
Cyclohexene......	82.5 to 83.5	0.8100	1.4465
Diisobutene......	101 to 102.5	0.7175 ±0.0015	1.4112

Procedure

7. (*a*) Place 10 ml. of carbon tetrachloride in a 50-ml. volumetric flask and, by means of a pipet, introduce a quantity of sample as indicated in Table II (Note 4). Either obtain the weight of sample introduced by difference between the weight (to the nearest milligram) of the flask before and after addition of sample or, if the density is known accurately, calculate the weight from the measured volume. Fill the flask to the mark with carbon tetrachloride and mix well (Note 5). Pipet a 5-ml. aliquot of the solution into a 500-ml. iodine number flask containing 50 ml. of glacial acetic acid.

NOTE 4.—Frequently, the order of magnitude of the bromine number of a sample is unknown. In this case, a trial test is recommended using a 2-g. sample in order to obtain the approximate magnitude of the bromine number. This exploratory test is to be followed with another determination using the appropriate sample size as indicated in Table II.

NOTE 5.—The dilution of the sample may be made with glacial acetic acid in place of carbon tetrachloride if desired. If the dilution of the sample is made with glacial acetic acid, the 5-ml. aliquot should be added to an iodine number flask containing 45 ml. of glacial acetic acid and 5 ml. of of carbon tetrachloride.

TABLE II.—SAMPLE SIZE.

Bromine Number	Sample Size, g.
0 to 10	20 to 16
Over 10 to 20	10 to 8
Over 20 to 50	5 to 4
Over 50 to 100	2 to 1.5
Over 100	1 to 0.5

(*b*) Shield the flask from exposure to direct sunlight and keep it at a temperature of 25 ± 5 C. Add bromide - bromate solution from a buret at a rate of 1 to 2 drops per second, swirling the flask constantly during the addition. Continue adding the reagent until the reaction mixture assumes a yellow color (Note 6) that remains for at least 5 sec. Add an additional 1 ml. of the reagent as quickly as possible, stopper the flask, and continue swirling immediately for 40 ± 5 sec. At the end of this period, place 5 ml. of KI solution in the lip of the flask and lift the stopper, allowing the solution to flow slowly into the flask. Replace the stopper, shake vigorously, add 100 ml. of water, and shake again for 1 min. Titrate promptly with $Na_2S_2O_3$ solution. Near the end of the titration, add 1 ml. of starch indicator solution and titrate slowly to disappearance of the blue color. Discard the test if the back-titration is less than 5 ml. or greater than 10 ml. of $Na_2S_2O_3$ solution (Note 7).

NOTE 6.—The yellow color produced should be equivalent to that obtained by adding 0.5 ml. of bromide - bromate solution to 50 ml. of glacial acetic acid and 5 ml. of carbon tetrachloride in a similar flask.

NOTE 7.—In certain cases, notably high-boiling samples, it is impossible to meet the requirements of a 5 to 10-ml. $Na_2S_2O_3$ back-titration; in such cases, the results are indeterminate.

(*c*) *Blanks.*—Make duplicate blank determinations on each lot of reagents by repeating the entire procedure but using 5 ml. of carbon tetrachloride in place of the sample (Note 8).

NOTE 8.—In routine work, no correction need be applied so long as the blank does not exceed 0.02 milliequivalent of bromine. For work of highest accuracy, a correction should be applied if the blank exceeds 0.01 milliequivalent. The correction shall be made by subtracting from the term $(VN - vn)$ in Section 8 the milliequivalents of bromine consumed by the volume of carbon tetrachloride actually present in the 5-ml. aliquot of sample solution used in the analysis. This volume may vary from 2.5 to 4.9 ml., depending upon the quantity of sample used.

Calculation

8. Calculate the bromine number of the sample as follows:

$$\text{Bromine number} = \frac{(VN - vn) \times 7.99}{W}$$

where:

V = milliliters of bromide - bromate solution added to the sample,

N = normality of the bromide - bromate solution,

v = milliliters of $Na_2S_2O_3$ solution used for the back-titration,

n = normality of the $Na_2S_2O_3$ solution, and

W = grams of sample represented by the aliquot used.

Precision

9. Results should not differ from the mean by more than the following amounts:

A.S.T.M. 90 per cent Distillation Point, deg. Fahr.	Range of Bromine Number	**Repeatability,** One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
Under 400	0 to 20	0.5	1
	Over 20 to 50	1	2
	Over 50 to 100	2.5	5
400 to 620	0 to 10	2	4
	Over 10 to 20	3	6

APPENDIX III

PROPOSED METHOD OF TEST FOR BROMINE NUMBER OF PETROLEUM DISTILLATES (ELECTROMETRIC METHOD)[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method is intended for the determination of the bromine number of petroleum distillates that are substantially free of material lighter than isobutane and that have 90 per cent distillation points under 620 F. The method is generally applicable to materials that fall within the following limits:

A.S.T.M. 90 per cent Distillation Point, deg. Fahr.	Bromine number, max.
Under 400	100
400 to 620	40

(*b*) Because of the variable reactivity of olefins, the bromine number obtained on various petroleum distillates is not an absolute measure of the olefins present but rather a criterion of certain properties of the material tested. It is known that high bromine numbers are obtained with polymer gasolines and those containing highly branched olefins or diolefins. Also, samples containing anthracene-type aromatics, sulfur, oxygen, and nitrogen compounds react to a certain extent with bromine under the conditions of the test. Thus, when bromine numbers are converted to olefins, the values obtained may not be definite and may vary from case to case unless the composition of the material tested is well established.

Definition

2. *Bromine Number* is the number of grams of bromine consumed by 100 g. of the sample when reacted under given conditions.

Outline of Method

3. The sample is dissolved in a specified solvent and titrated with bromide-bromate solution at 0 to 5 C. The end point is indicated by a dead-stop electrometric titration apparatus.

Apparatus

4. (*a*) *Dead-Stop Electrometric Titration Apparatus.*—The method as written is based on the use of the electric eye titrimeter described in the Supplement.[2] Any other dead-stop apparatus may be used with approximately 0.5 v. across the two platinum electrodes and capable of measuring the potential across the electrodes with a sensitivity of plus or minus 50 mv.

(*b*) *Titration Cell.*—A 150-ml. glass vessel jacketed to permit cooling with ice water shall be used. The pair of platinum wire electrodes shall be mounted to extend well below the liquid level. Stirring shall be by means of a mechanical stirrer and shall be rapid but not so vigorous that air bubbles are drawn down to the electrodes.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, October 1949; Revised, 1950.

[2] An electric eye titrimeter conforming to these requirements may be obtained from the Berkeley Scientific Co., Richmond, Calif. A Beckman Model H-2 pH meter, with adapter No. 700-1, has also been found satisfactory for this purpose.

Reagents

5. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. References to water shall be understood to mean distilled water.

(*a*) *Titration Solvent.*—Prepare 1 liter of titration solvent by mixing the following volumes of materials: 714 ml. of glacial acetic acid, 134 ml. of carbon tetrachloride, 116 ml. of methanol, 18 ml. of H_2SO_4 (1:5), and 18 ml. of a methanol solution of $HgCl_2$ (100 g. per l.).

(*b*) *Bromide - Bromate Solution* (*Standard 0.500 N*).—Dissolve 51.0 g. of KBr and 13.92 g. of $KBrO_3$ in water and dilute to 1 liter in a volumetric flask.

Check Procedure

6. In case of doubt in applying the procedure to actual samples, the reagents and techniques should be checked by means of determinations on freshly purified cyclohexene and diisobutene. Make the tests according to the procedure described in Section 7, using as a sample either 1.5 to 2 g. of freshly purified cyclohexene or diisobutene (see Table II) or 15 to 20 g. of 10 per cent by weight solutions of these materials in carbon tetrachloride. If the reagents and technique are correct, values within the following ranges should be obtained.

Standard	Bromine Number
Cyclohexene, purified (Notes 1 and 2)	191 to 199 (Note 3)
Cyclohexene, 10 per cent solution	19 to 20
Diisobutene, purified (Notes 1 and 2)	142 to 148 (Note 3)
Diisobutene, 10 per cent solution	14 to 15

NOTE 1.—Purified samples of cyclohexene and diisobutene may be prepared from Eastman cyclohexene, No. 1043, and Eastman diisobutene, No. P2125, by the following procedure:

Add 65 g. of activated silica gel (28 to 200 mesh)[3] to a column approximately 16 mm. in inside diameter and 760 mm. in length, which has been tapered at the lower end and which contains a small plug of glass wool at the bottom. A 100-ml. buret, or any column that will give a height-to-diameter ratio of the silica gel of at least 30:1, will be suitable. Tap the column during the addition of the gel to permit uniform packing.

To the column add 30 ml. of the olefin to be purified. When the olefin disappears into the gel, fill the column with methanol. Discard the first 15 ml. of percolate and collect the next 10 ml., which is the purified olefin for test of the bromine number procedure. Determine and record the density and refractive index of the purified samples at 20 C. Discard the remaining percolate.

Caution.—If distillation of these olefins is employed as a pre-purification step, at least 10 per cent residue should remain in the distillation flask to minimize the hazards from decomposition of any peroxides that may have been present.

NOTE 2.—The reference olefins yielding the above results are characterized by the properties shown in Table I.

NOTE 3.—The theoretical bromine numbers of cyclohexene and diisobutene are 194.8 and 142.3, respectively.

TABLE I.—PHYSICAL PROPERTIES OF PURIFIED OLEFINS.

Compound	Boiling Point, deg. Cent.	Density at 20 C., g. per cu. cm	Index of Refraction, *D* Line at 20 C.
Cyclohexene	82.5 to 83.5	0.8100	1.4465
Diisobutene	101 to 102.5	0.7175 ±0.0015	1.4112

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[3] Silica gel satisfactory for this purpose can be obtained from the Davison Chemical Corp., Baltimore, Md.

Procedure

7. (*a*) Place 10 ml. of carbon tetrachloride in a 50-ml. volumetric flask and, by means of a pipet, introduce a quantity of sample as indicated in Table II (Note 4). In the case of samples having bromine numbers in the range of 0 to 1, it is recommended that a 5-g. sample with 5 ml. of carbon tetrachloride or chloroform as a solvent be added directly to the titration vessel. Either obtain the weight of sample introduced by difference between the weight (to the nearest milligram) of the flask before and after addition of sample or, if the density is known accurately, calculate the weight from the measured volume. Fill the flask to the mark with carbon tetrachloride and mix well. Pipet a 5-ml. aliquot of the solution into the titration vessel containing 110 ± 10 ml. of the stirred titration solvent cooled to 0 to 5 C.

NOTE 4.—Frequently, the order of magnitude of the bromine number of a sample is unknown. In this case, a trial test is recommended using a 2-g. sample in order to obtain the approximate magnitude of the bromine number. This exploratory test is to be followed with another determination using the appropriate sample size as indicated in Table II.

TABLE II.—SAMPLE SIZE.

Bromine Number	Sample Size, g.
0 to 1	50
Over 1 to 25	20
Over 25 to 100	5
Over 100	2

(*b*) Adjust the "eye" of the electrometric titration apparatus to a very nearly closed position. Add the bromide-bromate solution in small increments from a buret, noting the action of the "eye," which when open shows the presence of unabsorbed bromine. The end point is indicated when two drops of reagent cause the "eye" to remain open for at least 30 sec.

(*c*) *Blanks.*—Make duplicate blank titrations on each lot of titration solvent and reagents by repeating the entire procedure, using 5 ml. of carbon tetrachloride in place of the sample. Less than 0.1 ml. of bromide-bromate solution should be required.

Calculation

8. Calculate the bromine number as follows:

$$\text{Bromine number} = \frac{(A - B)N \times 7.99}{W}$$

where:

A = milliliters of bromide - bromate solution used for titration of the sample,
B = milliliters of bromide-bromate solution used for titration of the blank,
N = normality of the bromide-bromate solution, and
W = grams of sample used.

Precision

9. Results should not differ from the mean by more than the following amounts:

A.S.T.M. 90 per cent Distillation Point, deg. Fahr.	Range of Bromine Number	**Repeatability,** One Operator and Apparatus	**Reproducibility,** Different Operators and Apparatus
Under 400	0 to 20	0.7	1.5
	Over 20 to 50	2	4
	Over 50 to 100	2.5	5
400 to 620	0 to 10	1	2
	Over 10 to 20	2	4

SUPPLEMENT

APPARATUS

Apparatus:

A1. The wiring diagram of the recommended electrometric titration apparatus is shown in Fig. 1. Connection to the platinum wire electrodes is made at points E_1 and E_2 in the upper left-hand portion of the diagram.

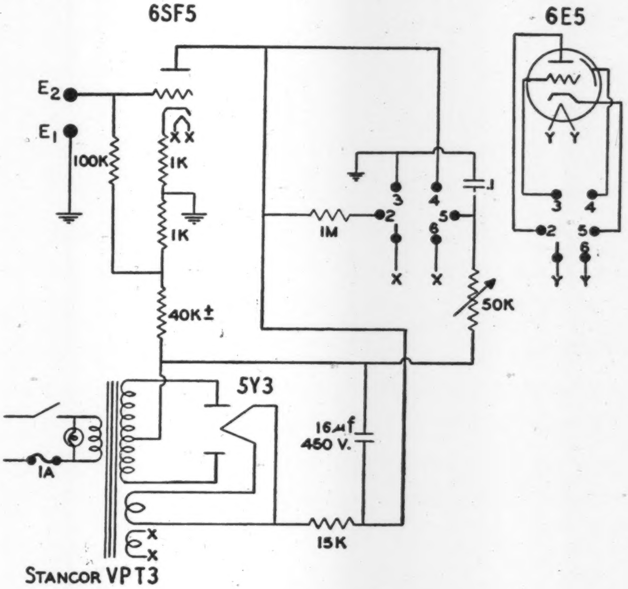
6SF5
6E5
E2
E1
100K
XX
1K
1K
40K ±
1M
3
4
2
5
6
X
X
.1
50K
Y Y
3
4
2
5
6
Y
Y
5Y3
16µf
450 V.
1A
15K
X
X
STANCOR VPT3

FIG. 1.—Electrometric Titrimeter.

APPENDIX IV

PROPOSED METHOD OF TEST FOR MEASUREMENT OF REFRACTIVE INDEX AND REFRACTIVE DISPERSION OF HYDROCARBON LIQUIDS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the measurement of refractive indices, accurate to six units in the fifth decimal place, and refractive dispersions, accurate to twelve units in the fifth decimal place, of transparent and light-colored hydrocarbon liquids which have refractive indices in the range between 1.33 and 1.50, and at temperatures from 68 to 86 F. (20 to 30 C.). The method is not applicable within the accuracy stated to liquids having colors darker than Union Color No. 4, to liquids having bubble points so near the test temperature that a reading cannot be obtained before substantial weathering takes place, to liquids having a refractive index above 1.50, or to measurements made at temperatures above 86 F. (30 C.).

NOTE 1.—The instrument can be successfully used for refractive indices above 1.50, and at temperatures both below 68 F. (20 C.) and above 86 F. (30 C.), but as yet certified liquid standards for the ranges above a refractive index of 1.50 are not available, so the precision and accuracy of the instrument under these conditions have not been evaluated. Similarly, certified refractive indices of liquids at temperatures other than the 68 to 86 F. (20 to 30 C.) range are not available, although the instrument can be used up to 122 F. (50 C.).

Definitions

2. (*a*) *Refractive Index* is defined as the ratio of the velocity of light (of specified wave length) in air, to its velocity in the substance under examination. It may also be defined as the sine of the angle of incidence divided by the sine of the angle of refraction, as light passes from air into the substance. This is relative index of refraction. If absolute refractive index (that is, referred to vacuum) is desired this value should be multiplied by the factor 1.00027, the absolute refractive index of air. The numerical value of refractive index of liquids varies inversely with both wave length and temperature.

(*b*) A *Refractive Dispersion* of a substance is the difference between its refractive indices for light of two different wave lengths, both indices being measured at the same temperature. For convenience in calculations, the value of the difference thus obtained is usually multiplied by 10,000.

Outline of Method

3. The refractive index is measured by the critical angle method with a Bausch and Lomb Precision Refractometer using monochromatic light. The instrument is previously adjusted by means of a solid reference standard and

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1950.

the observed values are corrected, when necessary, by a calibration obtained with certified liquid standards.

Apparatus

4. (*a*) *Refractometer*, Bausch and Lomb, "Precision" type,[2] range 1.33 to 1.64 for the sodium D line.

(*b*) *Thermostat and Circulating Pump*, capable of maintaining the indicated prism temperature constant within 0.04 F. (0.02 C.) of the desired test temperature. The thermostating liquid should pass the thermometer on leaving, not on entering, the prism assembly.

(*c*) *Thermometer*, graduated to permit readings to ±0.02 F. (0.01 C.). The bore correction at the temperature of test and under the emergent stem conditions encountered in use must be known to ±0.02 F. (0.01 C.).

(*d*) *Light Sources*.—The following light sources have been found satisfactory:

(*1*) *Sodium Arc Lamp*.—The Unitized "Sodium Lab Arc" is furnished with the instrument.

(*2*) *Mercury Arc Lamp*.—The H-4 type capillary mercury arc is furnished as an accessory to the refractometer.

(*3*) *Hydrogen Discharge Lamp*.—Any type lamp capable of producing light having an intensity of at least 3-foot candles on an area of one square centimeter on the entrance face of the illuminating prism. The luminous intensity may be conveniently measured by means of a photographic light meter held 10 in. from the lamp and perpendicular to the light beam. For convenience, the lamp should be mounted on an extension of the sodium lamp support.

(*4*) *Other Sources*.—Helium may be used in place of hydrogen in the lamp discussed under item (3).

(*5*) *Light Filters*.—For isolating the various spectral lines from the above sources, special light filters are required. The following are tentatively recommended:

Wave Length, Angstroms	Spectral Line	Filter
6678	Helium	Corning No. 2404
6563	H_C	None required. May use Corning No. 2404.
5893	Na_D	None required.
5461	Hg_e	Wratten No. 62, Corning Nos. 3486 + 4303 + 5120
5016	Helium	Wratten No. 45
4861	H_F	Corning Nos. 5030 + 3387, 4303, or Wratten No 45
4358	Hg_g	Corning Nos. 5113, 3389 + 5850.

NOTE 2.—In determinations of refractive indices above approximately 1.53 (wherever the short wave lengths show a higher scale reading than the long) this system of filters is rendered worthless and filters must be chosen which remove all spectral lines of shorter wave length than the one being read. Below this refractive index, the specific filters listed above, which remove spectral lines of longer wave lengths than the one being read, should be used.

Solvents

5. (*a*) *Ligroine*, end point not over 176 F. (80 C.) as determined by A.S.T.M. Method D 86, Test for Distillation of Petroleum Products.[3]

(*b*) *Benzene*, conforming to A.S.T.M. Specifications D 361, for Industrial 90 Benzene.[3]

Reference Standards

6. (*a*) *Solid Reference Standard* accurate to ±0.00002 with the value of the refractive index engraved upon its upper face.

(*b*) *Primary Liquid Standards*.—The organic liquids listed below, with the values of their refractive indices for the

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[2] Bausch and Lomb Optical Co., Rochester, N. Y., Catalogue No. 33-45-03. All instrument terminology used in this method corresponds with that used in the "Reference Manual" supplied with the instrument.

[3] 1949 Book of A.S.T.M. Standards, Part 5.

D, *F*, and *C* lines certified at 68, 77, and 86 F. (20, 25, and 30 C.), obtained from the National Bureau of Standards:

2,2,4-Trimethylpentane	$n_D = 1.39$
Methylcyclohexane	$n_D = 1.42$
Toluene	$n_D = 1.49$

Sample

7. A sample of at least 0.5 ml. is required. The sample shall be free of suspended solids, water, or other materials that tend to scatter light. Water may be removed from hydrocarbons by treatment with calcium chloride followed by filtering or centrifuging to remove the desiccant. The possibility of changing the composition of a sample by action of the drying agent, by selective adsorption on the filter, or by fractional evaporation, shall be considered.

Preparation of Apparatus

8. (*a*) The refractometer shall be kept scrupulously clean at all times. Dust and oil if allowed to accumulate on any part of the instrument will find its way into the moving parts, causing wear and eventual misalignment; if permitted to collect on the prism, dust will dull the polish, resulting in hazy lines.

(*b*) Thoroughly clean the prism faces with a cotton swab saturated with a suitable solvent, such as benzene, using surgical-grade absorbent cotton. Pass the swab very lightly over the surface until it shows no tendency to streak. Repeat this procedure with ligroine until both the glass and the adjacent polished metal surfaces are clean. Do not dry the prism faces by rubbing with dry cotton.

(*c*) Adjust the thermostat so that the temperature indicated by the refractometer thermometer is within 0.04 F. (0.02 C.) of the desired value; turn on the sodium vapor lamp and allow it to warm up 30 min.

NOTE 3.—An error of 0.04 F. (0.02 C.) in temperature of the sample will cause an error of 1×10^{-5} in the refractive index of methylcyclohexane.

(*d*) Control the ambient temperature within 2 F. (1 C.) of the test temperature. This can be done by regulation of the room temperature or by placing the instrument inside a specially designed constant-temperature box. The instrument shall also be so situated that it will not be subject to drafts.

Standardization of Apparatus and Technique

9. (*a*) Thoroughly clean the prism faces and surfaces of the solid reference standard as described in Section 8 (*b*), finally brushing the surfaces with a clean camels hair brush. Fix the hinged part in a wide-open position. Apply a drop of monobromonaphthalene, about 1.5 mm. in diameter, to the center of the polished surface of the reference standard. Press the reference standard against the surface of the stationary prism with the polished end toward the light. If the proper amount of contacting liquid has been used a continuous film of liquid will form between the prism and the reference standard, and the field will appear evenly illuminated. If not, irregular dark spots will appear in the illuminated field of the telescope when the knurled knob is turned and the light is in line with the longitudinal axis of the telescope. Gently manipulate the reference standard by pressure on one edge or another until the interference bands, as seen with the aid of the auxiliary lens, appear to extend horizontally in the rectangular contact area. It is well to keep the liquid wedge at such an angle that three to five bands can be seen and the fringe pattern should appear centered in the exit pupil of the telescope.

NOTE 4.—If there is any trace of roughness as the contact is being made, remove the reference standard and clean all surfaces again. More damage can be done to the prism surface in this operation than in weeks of use with liquids, if grit comes between the two surfaces during this contact. The amount of liquid should be just enough to fill the contact area completely, leaving no liquid at the front edge of the reference standard.

(*b*) Set the instrument to the scale reading corresponding to the refractive index engraved on the solid reference standard. Rotate the sodium lamp base while viewing the telescope until a sharp vertical line appears in the illuminated field and does not move with the rotation of the lamp. Adjust the eyepiece of the telescope to bring the cross hairs into sharp focus.

(*c*) Move the alidade by means of the hand wheel until the critical line on the left side of the band intersects the cross hairs, and read the scale. Repeat the setting at least twice, and between settings, shift the lamp slightly while observing the critical line in order to make sure a false line is not being observed. Average the scale readings for all the settings.

(*d*) Convert the average scale reading to refractive index by means of the table for the sodium *D* line. To give correct readings, without application of corrections, the average value obtained may not differ from that engraved on the test piece by more than 0.00002.

(*e*) If adjustment is necessary, set the scale to the reading corresponding to the value engraved on the solid reference standard, by means of the hand wheel on the side of the instrument. If the critical line is to the left of the intersection of the cross hairs, loosen the small screw on the left of the telescope and slowly tighten the one on the right until the lines coincide; if the critical line is to the right of the intersection, use the opposite procedure. At the final adjustment both screws should be snug but not tight. Again check the setting as in Paragraph (*c*).

Standardization with Reference Liquids

10. (*a*) Measure the refractive indices of each of the primary liquid standards listed in Section 6 (*b*) for the *D*, *F*, and *C* lines, at the test temperature 68, 77, or 86 F. (20, 25, or 30 C.), following the procedure described in Section 11. If the values obtained do not agree with the certified values within 0.00003, determine a correction curve for each wave length from an average of five independent determinations on each of the three certified liquid standards. A plot of the average error against refractive index provides a correction for all observed indices between these points.

NOTE 5.—This does not infer that the refractive index engraved on the test piece is necessarily inaccurate but tends to correct an error introduced in the determination by the failure to obtain grazing incidence in the case of liquid samples. This fault, and other instrumental errors, if present, are inherent in the refractometer design and its magnitude varies with the refractive index of the liquid and different instruments.

(*b*) To observe any changes with time and use in the relative positions of prism and alidade, each operator shall check the instrument with the calibrated solid reference standard prior to his use of the instrument.

Procedure

11. (*a*) Thoroughly clean the prism faces as described in Section 8 (*b*). Adjust the thermostat so that the temperature indicated by the refractometer thermometer is within 0.04 F. (0.02 C.) of the desired value.

(*b*) In testing nonviscous liquid samples, close the prism box and let stand for 3 to 5 min. to insure temperature equilibrium between the prisms and the circulating water. By means of a small pipet or medicine dropper, introduce a small quantity of sample into the tubulation between the prism faces. Turn the knurled head at the base of the telescope so as to bring the auxiliary lens into the light path, and observe through the face of the working prism. If the space between the prisms is completely filled with liquid, the field will be uniformly illuminated; bubbles or unfilled spaces will appear black. If the space is not completely filled, open the prism box slightly several times and add more liquid. Do not attempt to measure refractive indices until the space between the prisms is completely filled.

(*c*) In testing viscous liquids, open the prism box and apply the sample to the faces of both prisms, spreading evenly with a round wooden applicator stick. Never use metal or glass for this purpose as these may scratch the prism faces. Close the prism box slowly to avoid straining the hinge and locking mechanism.

(*d*) Adjust the illuminant to be in line with the telescope and bring the border line approximately to the reticle. While viewing the rear prism face by means of the auxiliary lens, rotate the lamp bracket to the right until only the extreme left side of the prism appears to be illuminated. If this rotation is carried too far vertical interference lines will appear in the back face. These are generally irregular and rather faint. The best adjustment for contrast and illumination seems to be the point just before these fringes become distinct.

(*e*) Adjust the eye piece of the telescope so as to bring the cross hairs into sharp focus, set the cross hairs on the critical edge and read the scale of the instrument. Readjust the position of the vapor lamp and repeat at least four times, approaching from either side of the critical edge and record the average scale reading. (In order to avoid the possibility of using a false edge, it is best to adjust the position of the light source each time a setting is made rather than make four settings on one positioning of the lamp.)

(*f*) Without changing the position of the prism assembly, place other desired light sources into the angular position (with respect to the rear face of the refracting prism) occupied by the sodium lamp. Take average scale readings for the desired lines in the manner described in Paragraph (*d*).

(*g*) In testing volatile samples, clean the prism faces without changing the position of the prism assembly or the lamp, recharge with sample, and read immediately.

Calculation and Reporting

12. (*a*) Convert the observed scale readings to refractive indices by use of the tables supplied with the instrument and report these values and temperature at which the test was made, distinguishing between the various spectral lines used (for example, "$n_D^t = 1 \ldots$" or "$n_{5893}^t = 1 \ldots$").

(*b*) To obtain refractive dispersion, subtract n_{λ_2} from n_{λ_1}. Report the result and the temperature at which the test was made (for example, "$(n_F - n_C) \times 10^4$ at $t = \ldots$" or "$(n_g - n_D) \times 10^4$ at $t = \ldots$").

Precision and Accuracy

13. (*a*) Results should not differ from the mean by more than the following amounts:

	Repeatability One Operator and Apparatus	Reproducibility Different Operators and Apparatus
Refractive Index	0.00006	0.00006
Refractive Dispersion	0.00012	0.00012

(*b*) Results should not differ from the true value by more than the following amounts:

	Accuracy
Refractive Index	0.00006
Refractive Dispersion	0.00012

APPENDIX V

PROPOSED METHOD OF TEST FOR MEASUREMENT OF DENSITY OF KNOCK TEST STANDARD NORMAL HEPTANE AND *ISO*OCTANE BY THE PYCNOMETER[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3. Pa.

Scope

1. This method is intended for the measurement of the density of pure hydrocarbons or petroleum distillates boiling between 194 and 230 F. (90 and 110 C.) that can be handled in a normal fashion as a liquid at the specified test temperatures of 68 and 77 F. (20 and 25 C.). It was developed especially for the reference fuels *n*-heptane and *iso*octane. It is designed to provide values having an accuracy of 0.00004 g. per ml.

Definition

2. *Density* is the weight in vacuo (that is, the mass) of a unit volume of the material at any given temperature. In this method the unit of mass is the gram, and the unit of volume the milliliter; the temperature scale used is the Centigrade.

Outline of Method

3. The liquid sample is drawn into the pycnometer and weighed. It is then equilibrated at the test temperature, and the positions of the liquid levels are observed. The density of the sample is then calculated from its weight and the volume occupied by the sample as determined by a calibration of the pycnometer with water at the same temperature. Buoyancy corrections are made in all weighings.

Apparatus

4. (*a*) *Precision Pycnometer*.—The pycnometer shall conform to the dimensions given in Fig. 1, be constructed of heat-resistant glass,[2] and have a total weight not exceeding 30 g.

(*b*) *Constant Temperature Bath*.—The water bath shall have a depth of at least 12 in., and be provided with means for maintaining a temperature of 68 ± 0.02 F. (20 ± 0.01 C.) or 77 ± 0.02 F. (25 ± 0.01 C.)

(*c*) *Bath Thermometer*.—A.S.T.M Kinematic Viscosity Thermometers 44 F. and 45 F. designed for tests at 68 F. (20 C.) and 77 F. (25 C.) and conforming to the requirements prescribed in A.S.T.M. Specifications E 1[3] may be used.[4] Ice point and bore corrections shall be known to the nearest 0.02 F. In use, the thermometers shall be immersed to a point at least 2 F. (1 C.) above the test temperature.

(*d*) *Pycnometer Holder*.—Figure 2 shows the structural details of the pycnometer holder. It may be made of brass or any other available metal that can be hard- or soft-soldered and that will not corrode in the thermostat liquid. Figure 3 illustrates a convenient mounting for suspending the holders in the constant temperature bath. It consists of a brass bar $\frac{1}{8}$ in. in thickness by 1 in. in width, of a length suitable for the bath used, and with seven $\frac{9}{32}$-in. holes drilled $1\frac{1}{2}$ in. apart to accommodate the threaded ends of the holders. Two nuts support each holder and permit regulation of the depth of immersion of the pycnometers.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1950.

[2] Pyrex glass has been found satisfactory.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

[4] At the present time there are no A.S.T M. Centigrade thermometers available that are particularly suitable for this test.

(*e*) *Balance.*—A balance able to reproduce weighings within 0.1 mg. when carrying a load of 30 g. or less on each pan. The balance should be located in a room shielded from drafts and fumes and in which the temperature changes between related weighings (empty and filled pycnometer) do not cause a significant change in the ratio of the balance arms. Otherwise weighings must be made by the substitution method in which the calibrated weights and pycnometer are alternately weighed on the same balance pan. The same balance shall be used for all related weighings.

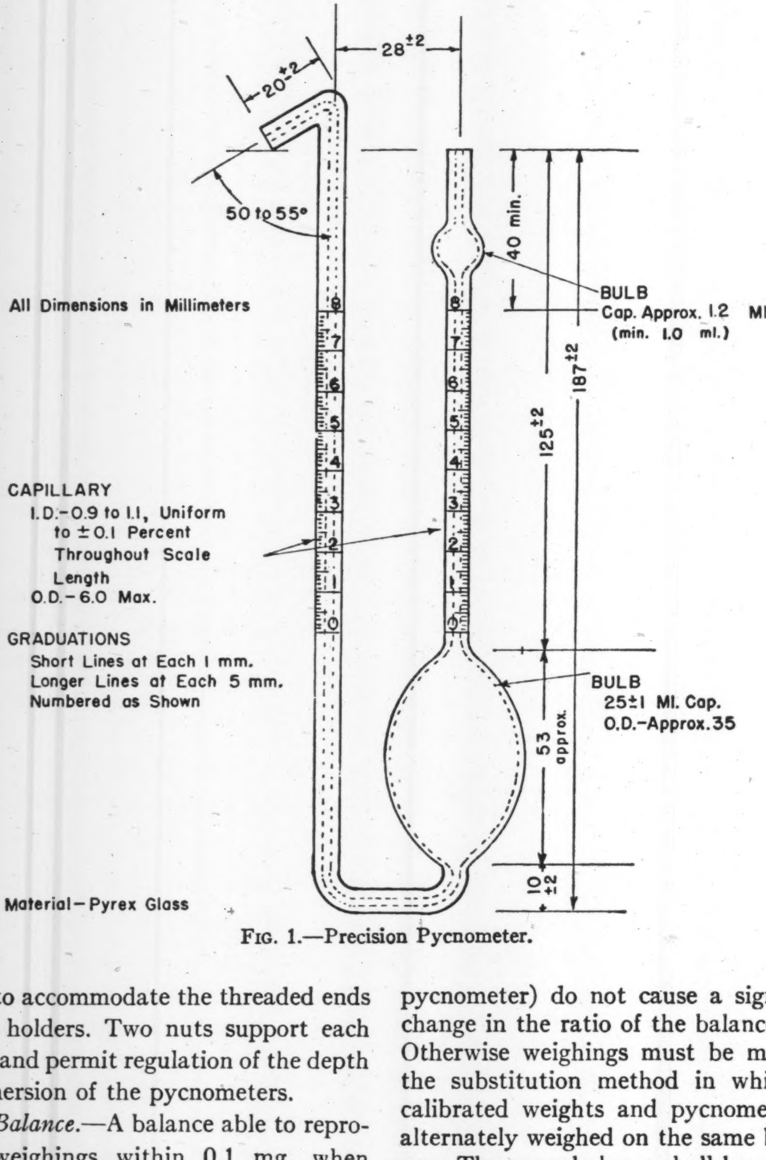
20±2
28±2
50 to 55°
40 min.
BULB
Cap. Approx. 1.2 Ml.
(min. 1.0 ml.)
All Dimensions in Millimeters
187±2
125±2
8 7 6 5 4 3 2 1 0
CAPILLARY
I.D.–0.9 to 1.1, Uniform
to ± 0.1 Percent
Throughout Scale
Length
O.D.–6.0 Max.
GRADUATIONS
Short Lines at Each 1 mm.
Longer Lines at Each 5 mm.
Numbered as Shown
BULB
25±1 Ml. Cap.
O.D.–Approx. 35
53 approx.
10 ±2
Material–Pyrex Glass

FIG. 1.—Precision Pycnometer.

(*f*) *Weights.*—Weights should be used whose relative values are known to the nearest 0.05 mg., or better. The same set of weights shall be used for the calibration of the pycnometer and the determination of the densities, or the sets of weights shall be calibrated relative to each other.

Preparation

5. Thoroughly clean the pycnometer, previous to calibration, by contact with chromic acid cleaning solution at room temperature. Rinse well with distilled water and dry at 190 to 200 F. (88 to 93 C.) for at least 1 hr., with a slow current of filtered air passing through the pycnometer, then cool to room temperature. Cleaning should be done in this manner whenever the pycnometer is to be calibrated or whenever liquid fails to drain cleanly from the walls of the pycnometer or its capillary. Ordinarily, the pycnometer may be cleaned between determinations by washing with a suitable solvent, such as isopentane or benzene, and vacuum drying. If the acetone is used as the wash liquid, the pycnometer should then be rinsed with isopentane or benzene.

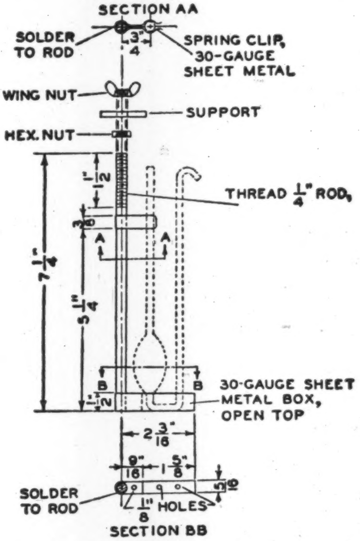

FIG. 2.—Pycnometer Holder.

Calibration of Apparatus

6. (*a*) Proceeding as directed in Section 7, determine the weight of freshly-boiled distilled water held by the pycnometer when equilibrated at the test temperature 68 or 77 F. (20 or 25 C.) with the water level at each of three different scale points on the graduated arms, two of which should be at opposite ends of the scale. Record this weight as W_{PW}.

(*b*) Calculate the true mass of the water M_W, in the pycnometer as follows (Note 1):

$$M_W = (W_{PW} - W_P)(1 + K)$$

where:

M_W = mass of water (weight of water corrected for buoyancy),

W_{PW} = weight of pycnometer plus water in air,

W_P = weight of pycnometer in air, and

K = buoyancy correction factor for

$$\text{water} = \frac{d_A}{d_W} - \frac{d_A}{d_w}$$

where:

d_A = density of air from Fig. 4 (Note 2),

d_W = density of water at 68 F. (20 C.) (0.99823 g. per ml.), or at 77 F. (25 C.) (0.99707 g. per ml.), and

d_w = density of weights (brass = 8.4).

NOTE 1.—It is assumed that the weighings of the pycnometer empty, and filled with water will be made in such a short time interval that the density of the air will not change significantly. If significant change should occur, then the procedure used in Section 8 (*b*) shall be followed.

NOTE 2.—The changes in humidity encountered in making a determination are seldom of sufficient magnitude to require correction. A 40 per cent change in humidity will cause a change of approximately 5 × 10^{-6} density unit. However, if unusual conditions prevail, the following equation, upon which the chart (Fig. 4) was based, may be used to calculate the air density.

$$D = \left[\frac{1.2929\,(273.16)}{T}\right]\left[\frac{B - 0.3783\,e\,H}{760}\right]10^{-3}$$

where:

D = density of air in grams per cu. cm.,

T = temperature in degrees absolute = (deg. Cent. + 273.16),

B = corrected barometric pressure in millimeters of mercury,

e = vapor tension of water at T in millimeters of mercury, and

H = relative humidity, expressed as a decimal.

(*d*) Prepare a calibration curve by plotting the sum of the scale readings in the two arms of the pycnometer against the corresponding true volume. If this curve is not a straight line, and subsequent checks do not correct the curvature, discard the pycnometer as imperfect.

(*e*) If a straight line cannot be drawn through the three points, determine enough additional points so that a straight line calibration may be drawn which does not lie more than 0.0002 ml. in units from the points used to determine the line.

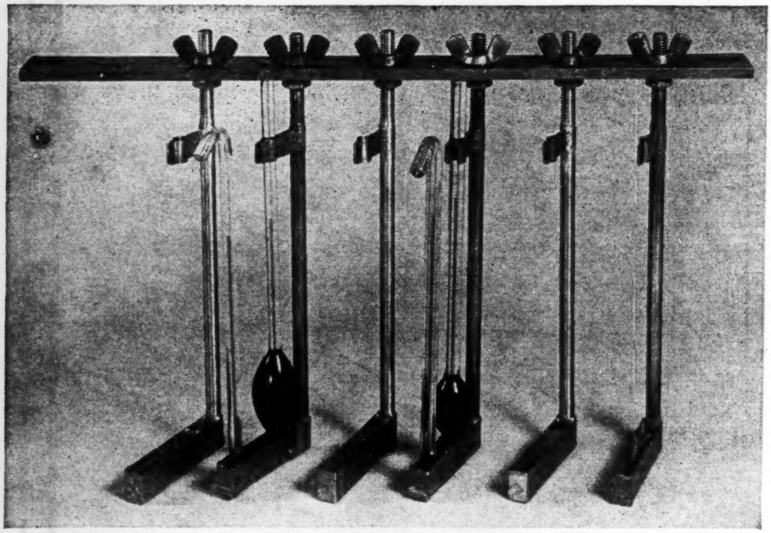

FIG. 3.—Holder Mounting.

(*c*) Determine the true volume of water in the pycnometer by the following equation:

$$V_t = \frac{M_W}{d}$$

where:

V_t = true volume of pycnometer at test temperature,

M_W = true mass of water in pycnometer, and

d = density of water at 20 C. (0.99823 g. per ml.), or at 25 C. (0.99707 g. per ml.).

Procedure

7. (*a*) This procedure is intended for pure compounds and mixtures that are not highly volatile, that is, which are essentially free of materials boiling less than 10 C. above the bath temperature.

(*b*) Using another pycnometer as a tare (Note 3), weigh the clean, dry pycnometer to 0.1 mg. and record the weight as W_P.

NOTE 3.—It is convenient to use the lightest of a set of pycnometers as a tare. For the best results the treatment and environment of both

pycnometer and tare for some time prior to weighing should be identical.

(*c*) Fill the pycnometer with the sample at approximately the test temperature by holding it in an upright position and placing the hooked tip in the sample, allowing the liquid to be drawn over the bend in the capillary by surface tension. Allow the pycnometer to fill by siphoning (requiring about 5 min.) and break the siphon when the liquid level reaches the graduation mark just above the large bulb, if filling is done at higher than but very close to bath temperature. The higher the filling temperature above bath temperature, the higher in the capillary should the liquid be allowed to rise (Note 4).

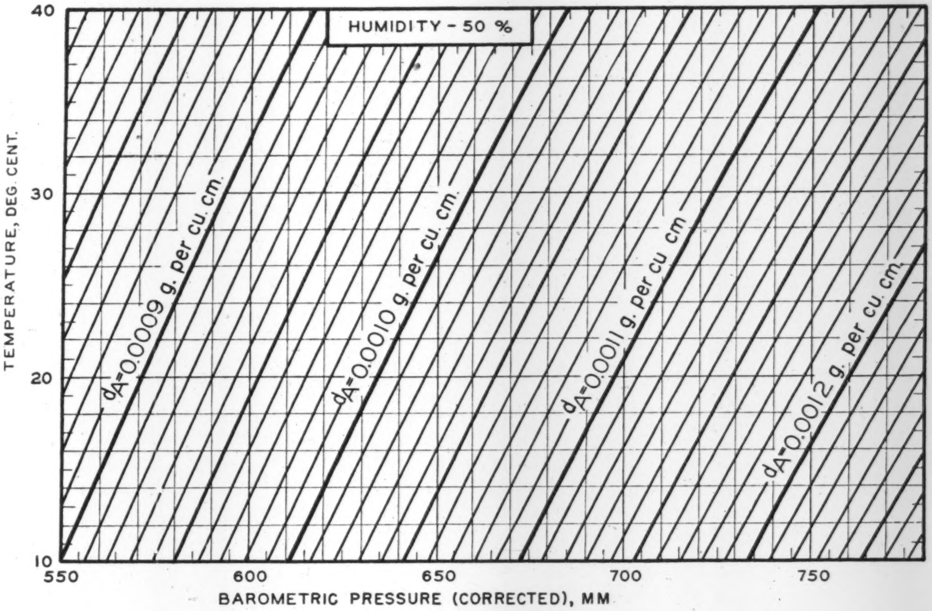

FIG. 4.—Air Density Chart.

NOTE 4.—These five-place densities can be determined most conveniently in a laboratory in which the room temperature and bath temperature are almost identical. If they are to be determined in a room where the temperature is 5 to 10 C. higher than bath temperature, the liquid should be pre-cooled to test temperature, the pycnometer filled to the desired level and placed in the bath long enough to come to equilibrium at the bath temperature, the scale readings recorded and the filled pycnometer removed from the bath, dried and weighed after allowing sufficient time for the sample to reach ambient temperature. If the pycnometer plus sample is weighed before the volume measurement, as required in the present procedure, liquid expands into the expansion bulb and incomplete drainage from the expansion bulb causes error in the volume measurement. This is more serious if it occurs during calibration with water.

(*d*) Wipe off the wet tip thoroughly (Note 5) with a chemically clean, lint-free cloth slightly damp with water and weigh to the nearest 0.1 mg. against the same tare and record the weight as W_{PS}. Record the temperature and barometric pressure at time of weighing.

NOTE 5.—In atmospheres of low humidity (60 per cent or lower) drying the pycnometer by rubbing with dry cotton cloth will induce static charges equivalent to a loss of about 1 mg. or more in the weight of the pycnometer. This charge may not be completely dissipated in less than ½ hr., and can be detected by touching the pycnometer to the wire hook on the balance and then drawing it away slowly. If the pycnometer exhibits an attraction for the wire hook, it may be considered to have a static charge. Radium bromide or polonium-coated foil in the balance case will dissipate the static charge.

(*e*) Place the pycnometer in the holder in a constant temperature bath adjusted to 68 or 77 ± 0.02 F. (20 or 25 ± 0.01 C.) Allow to remain for at least 30 min. or until two successive readings at 5-min. intervals show no change, and then read the scale to the nearest 0.2 small division at the liquid level in each arm. From the previously prepared calibration chart Section 6 (*d*), determine the true volume V_t.

Calculations

8. (*a*) Calculate the true density of the sample as follows:

$$\text{Density, g. per ml. at 68 or 77 F. (20 or 25 C.)} = \frac{M_S}{V_t}$$

where:

M_S = mass of sample as calculated in Paragraph (*b*), and

V_t = true volume of sample from Section 6 (*d*).

(*b*) If the pycnometer empty and the pycnometer containing the sample are weighed in air of the same density (within plus or minus 0.00001 g. per ml.), then calculate the mass of sample in the pycnometer as follows:

$$M_S = (W_{PS} - W_P)\,(1 + K)$$

where:

M_S = weight of the sample corrected for buoyancy, (that is, mass of the sample),

W_P = weight of the empty pycnometer in air,

W_{PS} = weight in air of the pycnometer and the sample contained in it, and

K = buoyancy correction factor for the liquid

$$= \frac{d_A}{d \text{ of sample (approx.)}} - \frac{d_A}{d_w}$$

where:

d_A = density of air, and

d_w = density of weights,

d = density in g. per ml.

Approximate density of sample is obtained by dividing the recorded weight of the sample in the pycnometer by its volume as determined from the calibration curve of the pycnometer.

$$\text{Approximate density} = \frac{W_{PS} - W_P}{V_t}$$

NOTE 6.—When the air density is appreciably different at the time of weighing the pycnometer plus sample (air condition II) than at the time of weighing the empty pycnometer (air condition I), the mass of sample in the pycnometer is calculated as follows: the weight of the pycnometer corrected for buoyancy is:

$$M_P = W_{PI}\,(1 + K_I)$$

where:

M_P = weight of empty pycnometer corrected for air buoyancy, (that is, mass of pycnometer),

W_{PI} = weight of pycnometer in air at density of condition I,

K_I = buoyancy correction factor at air condition I (using 2.2 for the density of glass), and

The weight in air, W_{PII}, of the pycnometer at the air density corresponding to a buoyancy correction factor K_{II} (air condition II) is:

$$W_{PII} = \frac{M_P}{1 + K_{II}}$$

Then complete the calculation as in Paragraph (*b*), substituting the value of W_{PII} for W_P.

Precision and Accuracy

9. (*a*) Results should not differ from the mean by more than the following amounts:

Repeatability One Operator and Apparatus	Reproducibility Different Operators and Apparatus
±0.00002	±0.00004

(*b*) Results should not differ from the true value by more than the following amounts:

Accuracy
±0.00004

APPENDIX VI

PROPOSED METHOD OF TEST FOR POUR STABILITY CHARACTERISTICS OF WINTER GRADE MOTOR OILS[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) This method describes the following two test cycles for estimating, in the laboratory, the field pour stability characteristics of winter grade motor oils having cloud points up to 40 F. (Note 1):

Cycle A (Fig. 1) for oil blends with cloud points in the temperature range of about 24 to 40 F.

Cycle C (Fig. 2) for oil blends with cloud points of about 24 F. and lower.

NOTE 1.—The winter grade motor oils include both regular and heavy duty, and diluted heavy duty motor oils. The A.S.T.M. cloud and pour point determination is described in A.S.T.M. Method D 97, Test for Cloud and Pour Points.[3]

(*b*) It is recommended that blends with high cloud points which show excellent pour stability characteristics by Cycle A, also be examined by Cycle C. For some of these blends Cycle C sometimes detects minor pour stability weaknesses not observed in Cycle A. For diluted oil blends, the cloud point before dilution should be used in deciding whether Cycle A or Cycle C is applicable.

(*c*) Motor oil blends with cloud points above 40 F. may also be examined by Cycle A, but for these blends there is a tendency for the laboratory stable pour points to be somewhat lower than field results. These blends may be examined in a special high-temperature test with over-all temperatures of the test roughly five degrees higher than Cycle A temperatures. Only in rare instances will cloud points of the test blends be above +40 F.

Outline Of Method

2. (*a*) *Cycle A.*—Oil blends are subjected to the following temperature sequence (Fig. 1) for six days: (*1*) rapid cooling from room temperature to about 14 F.; (*2*) warming to 34 F., this temperature being maintained for about 24 hr; (*3*) warming to about 50 F. followed by cooling to about +30 F. over a period of about 24 hr.; and (*4*) cooling from about +30 F. to −20 F. over a period of about two and one-half days.

(*b*) *Cycle C.*—Oil blends are subjected for six days to a temperature sequence quite similar to that of Cycle A but differing principally in that temperatures throughout the critical portions of the test are roughly 15 to 20 F. lower (Fig. 2). Samples are examined in part 4 of the cycle when the temperature reaches +20 F. and are re-examined at temperature intervals of five degrees until the minimum temperature of −20 F. is reached.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1950.

[2] McNab, Rogers, Michaels, and Hodges, "The Pour Stability Characteristics of Winter Grade Motor Oils," *Quarterly Transactions*, Soc. Automotive Engrs., Vol. 2, No. 1, p. 34, January, 1948. Hodges and Rogers, "Some New Aspects of Pour-Depressant Treated Oils," *Oil and Gas Journal*, October 4, 1947, p. 89.

[3] Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Apparatus

3. (*a*) *Cold Cabinet and Bath.*—A low-temperature cabinet capable of reducing oil sample temperatures to −25 F. shall be used. The bath consists of a copper tray placed on wooden supports, so that the bottom and sides are about 1 in. away from the bottom and sides of the interior of the cabinet, and containing a suitable liquid such as isopropyl or denatured ethyl alcohol (Note 2). Means shall be provided to maintain the bath at any desired temperature in the range of about +70 F. to −25 F.; normally, a heating coil is not necessary due to the slow warming rate. An automatic temperature controller shall be provided with cams profiled to reproduce the temperature sequences shown on the time-temperature charts, Figs. 1 and 2. The control bulbs shall be placed above the liquid near the top of the inner wall of the cabinet in order to obtain a steady rate of warming or cooling. In parts 2 and 3 of the temperature cycles, the temperature shall be controlled to plus or minus 3 F., and in part 3 the maximum temperature shall be between 48 and 52 F. for Cycle A, and between 32 and 35 F. for Cycle C (Note 3).

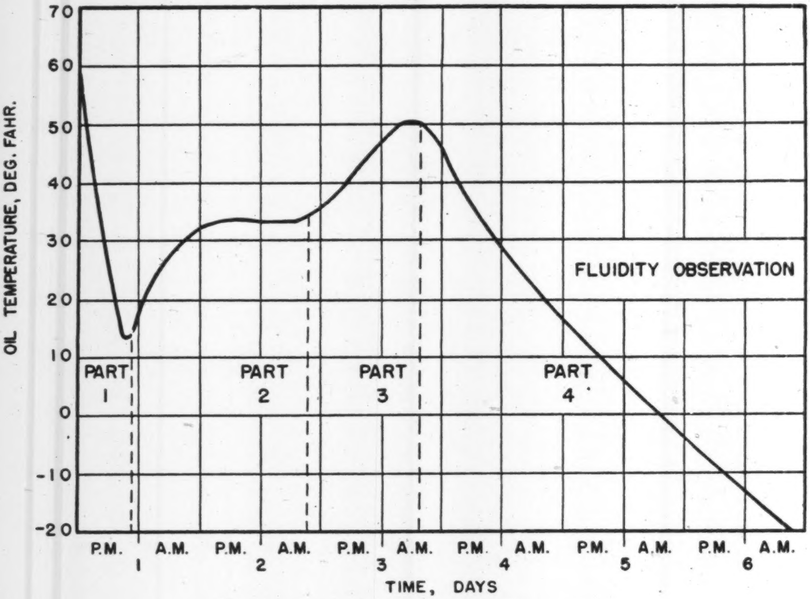

FIG. 1.—Cycle A Temperature Sequence.

NOTE 2.—The alcohol bath is used in place of an air bath to minimize fluctuations in oil temperature and to better maintain all test blends at the same temperature. A bath comprising a 50–50 mixture by volume of glycol and water may be used in place of the alcohol bath.

NOTE 3.—Some observers have noted that immersion of the temperature control bulb in the alcohol bath and agitation of the bath by a small air-driven agitator may give a temperature control of plus or minus 1 F.

(*b*) *Heating Water Bath.*—To preheat the test samples, a shallow bath of optional size and shape regulated to a temperature of about 130 F. shall be used. A bath 8 in. in depth and containing a wide meshed metal tray supported $1\frac{1}{2}$ in. from the bottom of the bath is adequate.

(*c*) *Temperature Measurement.*—The A.S.T.M. Cloud and Pour Thermometer 5F - 49, having a range of −36 to +120 F. and conforming to A.S.T.M. Specifications E 1[3] shall be used for measurement of the oil temperature. For obtaining a continuous record of the temperature of the cabinet and bath a double pen temperature recorder with one lead placed in the air near the control bulb, the other in a dummy sample in the bath liquid shall be used.

(*d*) *Sample Containers* shall be round, tall-form, 4-oz. bottles of clear flint glass.

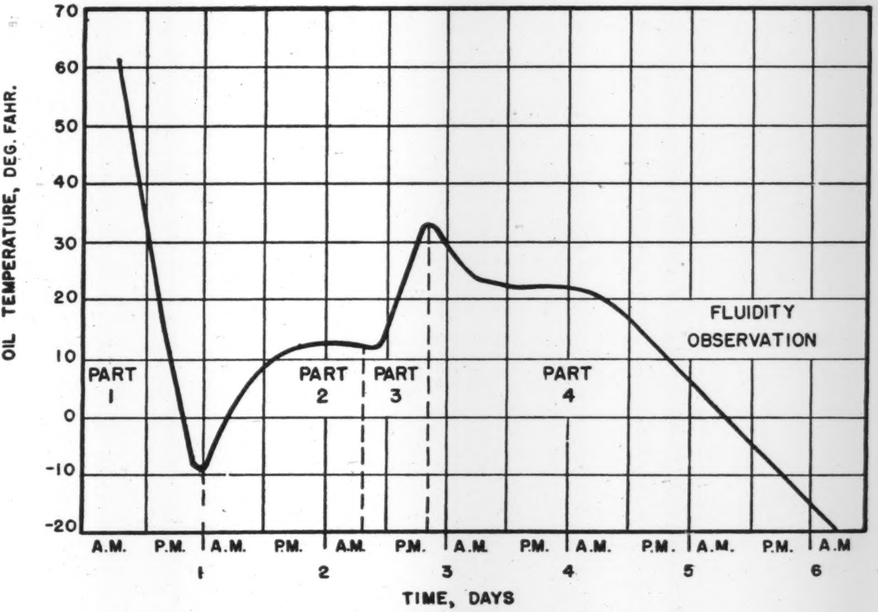

FIG. 2.—Cycle C Temperature Sequence.

Cloud Point Determination

4. Determine the cloud point of the sample in accordance with A.S.T.M. Method D 97, Test for Cloud and Pour Points.[3] If the sample is a diluted oil, determine the cloud point of the oil before dilution.

Preparation of Sample

5. (*a*) Fill the 4-oz. bottle half full with the sample and preheat in the heating bath to a temperature of about 115 F., with occasional but vigorous shaking. Keep it at this temperature for 10 min. After the preheating treatment, allow the sample to cool to room temperature (75 F.), then close the sample container with a clean, dry cork.

(*b*) Bring the cold cabinet to room temperature. If time permits, this may be done by leaving the cabinet open for 24 hr.; otherwise insert a steam coil.

(*c*) When both the sample and the bath are at room temperature, place the bottle in the bath. Place two dummy samples in the bath, one at either end. Insert one of the leads attached to the temperature recorder in one of the dummy samples, and place an A.S.T.M. Cloud and Pour Thermometer in the other with the immersion mark just visible, but not more than $\frac{1}{8}$ in. above the top of the stopper. Make all subsequent temperature observations on these dummy samples and not on the test sample. If necessary, add more bath liquid so that the fluid level will be maintained above that of the oil in the sample bottle. If numerous samples are being tested, it is important that at least ½ in. be left between each bottle.

Cycle A—Procedure

6. (*a*) Place the Cycle A cam guide, profiled in conformity with the time-temperature chart, Fig. 1, in the temperature control box and start the initial cooling (the cam guide is constructed from trial runs using manual controls to regulate the oil sample temperature as closely as possible in accordance with the recommended time-temperature charts). The actual temperatures will not exactly fit the smooth curves because of minor temperature fluctuations; but in order to obtain reproducible results maximum temperature fluctuations should be within plus or minus 3 F.

(*b*) During the final cooling period (part 4 of temperature cycle, Fig. 1), when the oil temperature reaches +20 F., and at every five-degree interval thereafter, remove the sample bottle from the bath, tilt it to an angle of 90 deg. from the vertical, hold there for 3 sec. and note the condition of the oil. Record solid points when no movement of the oil is observed after holding the sample bottle in a horizontal position for 3 sec. If the oil is very fluid, only a slight tilt will be necessary; do not allow the oil to come into contact with the stopper. Occasionally it may be noted toward the end of the test that when the bottle is tilted to the required angle, the surface may show no movement, but the whole core of solidified oil may slip forward very slightly. In this case consider the oil as solid; note only surface movement. Avoid agitation of the blends during the test as much as possible.

(*c*) Continue observations at intervals of five degrees until the surface of the oil shows no movement whatever when held in a horizontal position for 3 sec. (Note 4). Record the temperature of the sample at this point as the "highest solid temperature." If the oil is still fluid at −20 F., discontinue the test.

NOTE 4.—If at any time there is any doubt about a slight surface movement, return the sample to the bath and cool to the next lower five-degree interval. If the oil is definitely solid at the next lower interval, record the previous figure.

Cycle C—Procedure

7. Pretreat the sample as described in Section 5 and carry out the temperature sequence as described in Section 6 for Cycle A, except use the Cycle C cam guide, profiled in conformity with the time-temperature chart shown in Fig. 2.

Report

8. (*a*) Report the temperature 5 F. above the highest temperature at which the oil first becomes solid as "stable pour point, —— F., Cycle —."

(*b*) If the oil still shows surface movement at a temperature of −20 F., report the sample as "stable pour point, below −20 F., Cycle —."

(*c*) If a sample is tested by more than one cycle, report the results obtained by both cycles.

Precision

9. (*a*) *Repeatability.*—For oils with poor pour stability characteristics individual results on the same oil in any one laboratory may vary by 5 F. For blends with Cycle A solid points below 0 F. the results may vary by 10 F. in some cases (Note 5).

(*b*) *Reproducibility.*—Individual results of the test on the same oil in different laboratories may vary by 10 F. The average of three or more results in different laboratories should show a difference between averages no greater than 5 F.

NOTE 5.—As an aid in determining the validity of each individual test run, it is recommended that each test include two or more reference samples having different degrees of pour stability weakness and having stable pour points which have been well established.

APPENDIX VII

PROPOSED METHOD OF TEST FOR REDUCED PRESSURE DISTILLATION OF PETROLEUM PRODUCTS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method is intended for the determination, at reduced pressures, of the boiling temperature ranges of petroleum products which decompose when distilled at atmospheric pressure. The method is applicable, up to a maximum liquid temperature of 750 F. (400 C.), to the distillation of any petroleum product which starts to vaporize under the pressure of the test at a vapor temperature at least 50 F. (28 C.) above the condenser temperature used in the distillation.

NOTE 1.—Results by this method are not comparable with those of other A.S.T.M. procedures[2] for the determination of boiling ranges of petroleum products such as: A.S.T.M. Method D 86 for Gasoline, Naphtha, Kerosine, and Similar Petroleum Products, Method D 158 for Gas Oil and Similar Distillate Fuel Oils, Method D 216 for Natural Gasoline, Method D 285 for Crude Petroleum, Method D 447 for Plant Spray Oils, Method D 850 for Industrial Aromatic Hydrocarbons.

Outline of Method

2. The sample is distilled, at some predetermined and accurately controlled pressure, between 1 mm. of mercury, absolute, and atmospheric, under conditions which provide approximately one theoretical plate fractionation. Data are obtained from which a distillation curve can be prepared relating volume distilled and boiling point at the controlled pressure.

Apparatus

3. The apparatus is shown schematically in Fig. 1 and consists, in part, of the components described below. Other components of the assembled apparatus which appear in Fig. 1 are not specified, either as to design or as to performance. Some of these parts are not essential from the point of view of obtaining satisfactory results from the tests, but are desirable components of the assembly for the purpose of promoting the efficient use of the apparatus and ease of its operation.

(*a*) *Flask*, *Column*, *Condenser*, and *Product Receiver* shall conform to the dimensions and requirements given in the Supplement, Section A1.

(*b*) *Heater*, 750 w., constructed as described in the Supplement, Section A3, and provided with a suitable variable transformer for control of power input. Support the heater independently of the flask so that the heater can be readily lowered at the end of a distillation to hasten the cooling of the apparatus.

(*c*) *Thermocouple* for measurement of

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 5.

vapor temperature, constructed as described in the Supplement, Section A2.

(*d*) *Potentiometer* for use with the thermocouple referred to in Paragraph (*c*). The combined unit shall be capable of detecting temperature changes of 1 F. (0.6 C.) over the temperature range of 32 to 700 F. (0 to 370 C.).

(*e*) *Flask Thermometer or Thermocouple.*—An A.S.T.M. Partial Immersion Thermometer having a range of 20 to 760 F. and conforming to the requirements for thermometer 3 F - 49 as prescribed in A.S.T.M. Specifications E 1.[2] A thermocouple may be substituted for the thermometer, provided that the potentiometer used with it permits temperature readings with an accuracy equivalent to plus or minus 2 F. (1.1 C.).

(*f*) *Pressure Measuring System* capable of measuring absolute pressure with an accuracy of 1 per cent in the range of 1 mm. of mercury to atmospheric pressure. A manometer and McLeod gage which meet this requirement are described in the Supplement, Section A4 (*a*).

(*g*) *Pressure Regulating System* capable of maintaining the pressure of the system constant within plus or minus 1 per cent of the absolute pressure at 10 mm. of mercury or higher and within plus or minus 0.1 mm. of mercury at lower pressures. Suitable equipment for this purpose is described in the Supplement, Section A4 (*b*).

(*h*) *Safety Screen* that adequately shields the operator from the distillation apparatus.

Standardization of Apparatus

4. *Thermocouple.*—Calibrate[3] the thermocouple and potentiometer as a unit by comparison with a working standard in a stirred liquid bath. Use as calibration points the temperatures 32, 100, 200, 300, 400, 500, and 600 F. (0, 38, 93, 149, 204, 260, and 315 C.). If necessary for the potentiometer employed, prepare a chart by means of which the potentiometer reading can be converted to degrees Fahrenheit or Centigrade with an accuracy of 1 F. (0.6 C.).

NOTE 2.—The working standard may be either a platinum resistance thermometer or a calibrated mercury-in-glass thermometer certified by the National Bureau of Standards. A thermocouple calibrated by the National Bureau of Standards may be used as a working standard if the potentiometer, used to measure its emf., is accurate to plus or minus 0.02 mv.

Sample

5. The sample should be moisture free. When necessary, it may be dehydrated by any suitable means before it is weighed into the flask. A suggested method for dehydrating the sample is given in the Supplement, Section A5.

Preparation of Apparatus

6. (*a*) Mount the thermocouple in the column as shown in Fig. 3. The center line of the thermocouple must be coincident with the center line of the column, and the joint between the Kovar tip and the glass stem of the thermocouple must be on a level with the inside of the bottom of the vapor outlet tube at its junction with the column. The exact location of the tip of the thermocouple in the assembly is of the utmost importance.

(*b*) Connect the vacuum system and the manostat to the distillation apparatus through the connection at the top of the condenser. Connect the pressure measuring device to the tube at the top of the distillation column. Use a McLeod gage for pressures of 20 mm. of mercury and below; above 20 mm. of mercury use a manometer or other device having the specified accuracy.

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[3] For detailed directions for the calibration of thermocouples, see Wm. F. Roeser and H. T. Wensel, "Methods of Testing Thermocouples and Thermocouple Materials," *Journal of Research*, Nat. Bureau Standards, Vol. 14, 1935, pp. 247–282. (*RP*-768), or "Temperature, Its Measurement and Control in Science and Industry," A Symposium of the American Institute of Physics, Reinhold Publishing Corp., New York, N. Y., pp. 284–314 (1941).

NOTE 3.—It is desirable to install cold traps between the apparatus and the pressure measuring device and between the apparatus and the vacuum system, as indicated in Fig. 1. These are precautionary steps to protect the pressure measuring and controlling devices, but are not essential as far as the results of the test are concerned. A liquid nitrogen trap may be necessary if the sample being distilled contains much sulfur or releases corrosive gases. When a McLeod gage is used to measure the pressure, satisfactory operation of the gage requires that it be protected by a liquid nitrogen trap.

(*c*) Connect the circulating cooling fluid system so that the fluid flows, in order, through the distillate receiver jacket, the condenser, and the jacket of the side arm cooler. Adjust the temperature of the cooling fluid so that the liquid circulating in the system is at 140 ± 5 F. (60 ± 3 C.).

NOTE 4.—Although 140 F. (60 C.) is a suitable temperature for most distillations, other temperatures may be used, as required for convenient handling of the sample being distilled. In all cases, maintain the temperature of the cooling fluid in the system constant within plus or minus 5 F. (3 C.).

Procedure

7. (*a*) Determine the specific gravity of the oil sample at the temperature of the receiver by means of a hydrometer by A.S.T.M. Method D 287,[2] Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer, or other suitable means of equal accuracy.

(*b*) Weigh into the distillation flask a quantity of oil equivalent to 200 ml. of the sample at the temperature of the receiver. Determine the weight to within 0.1 g.

(*c*) Place a small plug of glass wool in the distillation flask thermometer well and insert the thermometer, or thermocouple.

(*d*) Lubricate the spherical joints of the distillation apparatus with a minimum amount of suitable grease. Make certain that the surfaces of the joints are clean before applying the grease and use only the minimum quantity required. Place the flask on the heater and connect the spherical joints of the apparatus together. Clamp the joints together by means of adjustable spring clamps.

NOTE 5.—Silicone high vacuum grease[4] has been used for this purpose. An excess of this lubricant applied to the flask joint may cause the sample to foam during distillation.

(*e*) Start the vacuum pump and observe the flask contents for signs of foaming. If the sample foams, allow the pressure on the apparatus to increase slightly until the foaming subsides. Gentle heat may be applied to assist the removal of dissolved gas. For general directions for suppression of excessive foaming of the sample, see the Supplement, Section A6.

(*f*) Evacuate the apparatus until the pressure reaches the level desired for the distillation. Failure to reach the distillation pressure, or an excessive increase in pressure in the apparatus with the pump shut off, is evidence of serious leakage into the system. Readmit air into the apparatus and relubricate all joints. If this does not result in a vacuum-tight system, examine other parts of the system for leaks by appropriate means.

(*g*) Turn on the heater and apply heat as rapidly as possible to the flask without causing undue foaming of the sample. As soon as vapor or refluxing liquid appears at the joint between the flask and the column, adjust the rate of heating so that the distillate is recovered at from 4 to 8 ml. per min. Continue the distillation until either a liquid temperature of 750 F. (400 C.) or the maximum vapor temperature is reached.

NOTE 6.—The maximum vapor temperature may result either from complete distillation of the oil before the flask temperature has reached 750 F. (400 C.) (that is, an end point) or because of the incidence of cracking. The latter will be evidenced by an increase in the distilling rate while the vapor temperature remains stationary or recedes. Adjustment of the distilling rate then results in a marked drop in vapor temperature. "Smoking," the presence of clouds of light vapor, and a significant increase in the pressure of the system are other indications of cracking. When these conditions are observed it is advisable to discontinue the distillation.

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[4] Manufactured by Dow Corning Corp., Midland, Mich.

(*h*) Lower the flask heater a few inches and cool the flask and heater with an air blast. Repressure the still with nitrogen, if it is necessary to dismantle the apparatus before it has cooled below 200 F. (93 C.). Carbon dioxide may also be used for repressuring, provided liquid nitrogen traps are not in use.

NOTE 7.—**Danger.**—Repressuring the still with air while it contains hot oil vapors may result in fire or explosion.

(*i*) Record the potentiometer reading which corresponds to the vapor temperature prevailing when each of the following volume percentage fractions of the charge has been collected in the receiver: 5, 10, 20, 30, 40, 50, 60, 70, 80, 90, 95, and at the end point. If the liquid temperature reaches 750 F. (400 C.), or if the vapor reaches a maximum temperature before the distillation end point is observed, record the potentiometer reading at the time the distillation is discontinued. In the latter case, record the total volume per cent recovered. When a product is tested for conformity with a given specification, make and record all necessary observations, whether or not they are included in the series listed in this paragraph.

(*j*) Observe the pressure indicated by the pressure measuring device on each occasion that a potentiometer reading is recorded. Record the observed pressure along with the corresponding vapor temperature for each observation.

Calculation and Report

8. (*a*) When necessary, convert the potentiometer readings to temperatures by means of the chart described in Section 4.

(*b*) Report the vapor temperatures in degrees Fahrenheit, corresponding to the volumetric percentages of liquid recovered at which the observations were made.

(*c*) Report the pressure under which the distillation was carried out. If the pressure fluctuates by more than 1 per cent of its absolute value during the distillation, report the actual pressure reading corresponding to each value of vapor temperature reported.

Precision

9. Cooperative work at 10 mm. of mercury indicated a repeatability and reproducibility of plus or minus 5 F. (3 C.) from the mean.

SUPPLEMENT

APPARATUS DESCRIPTION AND NOTES ON PROCEDURE

Distillation Apparatus Assembly

A1. (*a*) *Flask*, constructed of heat-resistant glass,[5] shall conform to the dimensions shown in Fig. 2. Insulate the flask with asbestos wicking and insulating cement as shown in Fig. 1.

NOTE 8.—A convenient means for insulating the flask is as follows:

> Wrap the flask with approximately 12 ft. of asbestos packing[6] in the following manner: Set the flask in the operating position, that is, in the hole in the transite board at the top of the heater assembly. Separate and flatten the strands of the packing for about 1 in. on one end. Starting at the top of the flask neck below the semiball joint, place the flattened end of the wicking on the neck. Start wrapping spirally, covering the flattened end with the first turn. Wrap tightly until reaching ⅜ in. from the transite board. Cut off the excess packing, leaving 1 in. of free end, flatten, and tuck it under the last turn.
>
> Wet the wicking on the flask by rubbing water into it. This prevents the cement which is applied later from drying too rapidly because of the absorption of water from the cement mixture.
>
> Thoroughly mix dry insulating cement[7] with approximately 5 per cent by volume of Portland cement. Add water to form a soft workable paste. Apply a ¼-in. layer of this mixture over all the wicking. Cover the upper and lower ends of the wicking so as to seal to the glass. Mold the cement into the space between the transite board and the wicking. Place the insulated flask in an oven and dry at 150 F. (65 C.).

(*b*) *Column*, constructed of heat-resistant glass,[5] shall conform to the dimensions shown in Fig. 3. Insulate the column with asbestos wicking and insulating cement as shown in Fig. 1. Install a foam breaker in the column as shown in Fig. 4.

NOTE 9.—A convenient means for insulating the column is as follows:

> Take 5 ft. of packing[6] and flatten one end. Place the flattened end on the column adjacent to the 19/38 standard tapered joint and wrap spirally, covering the flattened end with the first turn. Wrap tightly until the top of the vapor outlet tube is reached. Carry the packing to the other side of the vapor tube and continue wrapping the column to within ¾ in. of the male semiball joint. Cut off excess packing, leaving 1 in. of free end, flatten, and tuck it under the last turn.
>
> Take 4 ft. of packing, separate and flatten the end as before. Place the flattened end on the vapor outlet tube adjacent to the 35/20 spherical joint, and wrap spirally, covering the flattened end with the first turn. Wrap tightly until the uncovered portion of the column is reached. Then loop the wicking around the column, immediately above and below the side-tube. Cut off the excess packing, leaving 1 in. of free end, flatten and tuck it under the last turn. This should leave a space between wrappings opposite the vapor side-tube for a thermocouple sight hole.
>
> To provide for the thermocouple sight hole, place a No. 5 cork stopper with the small end next to the glass on the side opposite the vapor outlet connection. The center of the cork must be located in line with the inside of the bottom of the vapor outlet tube at its junction with the column.
>
> Wet all of the packing on the column and side-tube and cover with the insulating cement mixture, in a manner similar to that used for covering the distillation flask. Place the insulated column in an oven and dry at 150 F. (65 C.).

(*c*) *Condenser*, constructed of heat-resistant glass,[5] shall conform to the dimensions shown in Fig. 5. Provide a light link chain[8] for the condenser drip-tip as shown in Fig. 6.

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[5] Pyrex glass has been found satisfactory.

[6] The packing has an apparent diameter of 3/8 in. and weighs approximately 9 to 10 g. per foot. It consists of three loosely twisted strands of asbestos, each formed around a cotton string center. The requirements of this packing are met by Johns-Manville type No. 4202 asbestos wick packing.

[7] Johns-Manville No. 302 insulating cement is suitable.

[8] The types of chain used in the 200-g. "Chain-o-matic" balances (Christian Becker Co., Monhegan, Clifton, N. J.) or in the manufacture of costume jewelry are satisfactory.

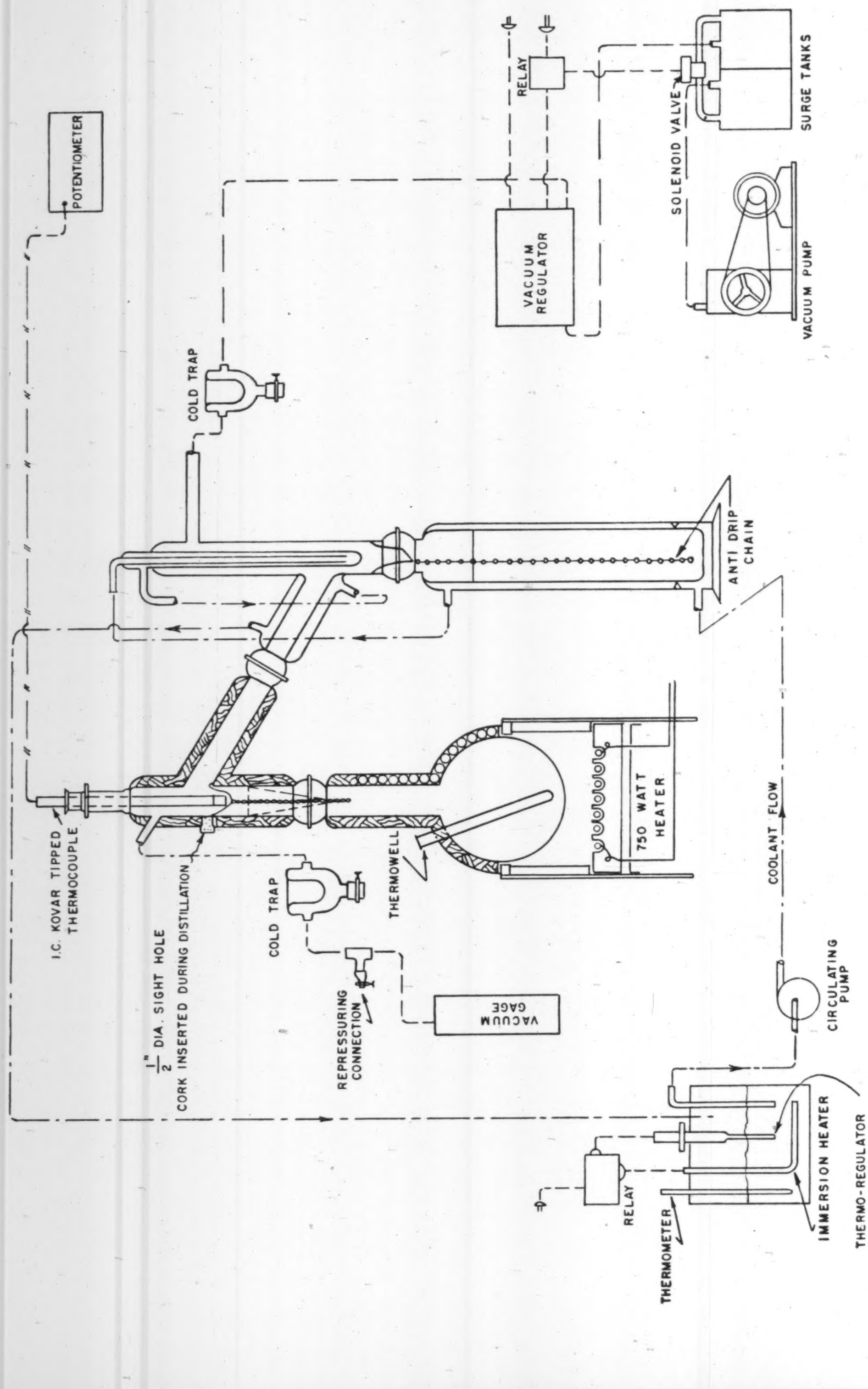
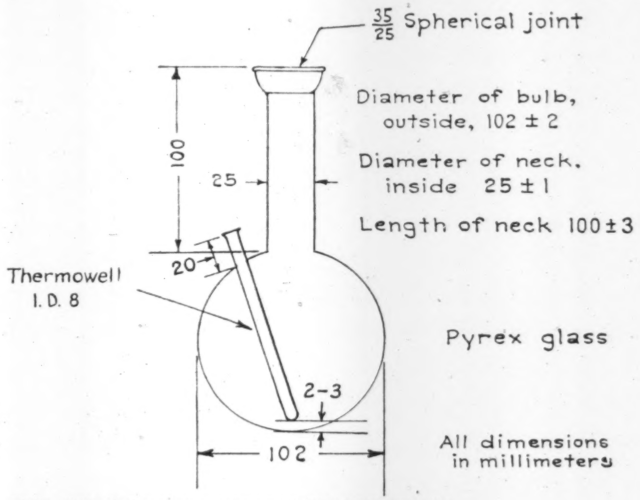

POTENTIOMETER
RELAY
SURGE TANKS
SOLENOID VALVE
VACUUM REGULATOR
VACUUM PUMP
COLD TRAP
ANTI DRIP CHAIN
I.C. KOVAR TIPPED THERMOCOUPLE
$\frac{1}{2}$" DIA. SIGHT HOLE
CORK INSERTED DURING DISTILLATION
THERMOWELL
750 WATT HEATER
COLD TRAP
COOLANT FLOW
REPRESSURING CONNECTION
VACUUM GAGE
CIRCULATING PUMP
RELAY
THERMOMETER
IMMERSION HEATER
THERMO-REGULATOR

FIG. 1.—Assembly of Vacuum Distillation Apparatus.

(*d*) ***Product Receiver***, constructed of heat-resistant glass,[8] shall conform to the dimensions shown in Fig. 7.

NOTE 10.—The glass parts of the apparatus are subjected to severe thermal conditions and, to lessen the chances of failure during a test, only equipment shown to be strain-free by a polarized light test should be used.


$\frac{35}{25}$ Spherical joint
Diameter of bulb, outside, 102 ± 2
Diameter of neck, inside 25 ± 1
Length of neck 100 ± 3
100
25
20
Thermowell I. D. 8
Pyrex glass
2-3
102
All dimensions in millimeters

NOTE: THE THERMOWELL MAY BE INSTALLED EITHER BY SEALING INTO THE FLASK OR BY MEANS OF A GROUND TAPERED JOINT. FOR DISTILLATIONS IN WHICH THE TEMPERATURE CANNOT EXCEED 750 F., THE THERMOWELL MAY BE OMITTED.

FIG. 2.—Distillation Flask.

Vapor Temperature Measurement

A2. *Thermocouple*,[9] iron-constantan, in Kovar tip, constructed as shown in Fig. 9.

Heater

A3. Heater shall be constructed as shown in Fig. 8.

Pressure System

A4. (*a*) *Pressure Measuring System*.—An appropriate closed-end mercury manometer and a McLeod gage comprise a suitable pressure measuring system. The manometer shall be long enough to measure all the pressures encountered in the use of the apparatus and shall be equipped with means for accurately measuring the differences in level of the arms under operating conditions. The McLeod gage shall have an accurately known volume ratio of at least 10 to 1 and be of the quadratic scale type. In any pressure measuring system adequate traps shall be provided to prevent vapors from the distillation apparatus from interfering with the functioning of the gages, or accidental flow of mercury or other manometric liquid into the manostat or distillation apparatus.

(*b*) *Pressure Regulating System*.—The following is suggested as a satisfactory example of a pressure regulating system: A "Pressovac" or similar low efficiency, high capacity pump is connected to one of two surge tanks, each having a capacity of 3 to 5 gal. and arranged in series. A solenoid valve or other type regulator is installed in the connection between the tanks so that the first tank is maintained at pump pressure and the second one at the pressure of the distillation apparatus. With some apparatus it may be desirable to have a slight bleed to the second tank which will cause the controls to operate at regular intervals in order to provide smooth operation. However, experience has shown that the bleed should be held at an absolute minimum in order to prevent loss of vapors through the manometer connection at the top of the column. Connecting lines from the second tank to the vacuum distillation apparatus should be as short in length and as large in diameter as possible. A diameter of ½ in. is suggested as a minimum for these connections. For multiple still arrangements, it is possible to use a large pump and a large low-pressure surge tank. Several smaller tanks operating at the pressures of the various distillations can be attached to the large low-pressure surge tank with individual pressure regulators. Other arrangements may be used, provided the pressure is maintained constant within the limits specified in Section 3 (*g*).

---

[9] The thermocouple used in this method has been described by Rolfson, Penther, and Pompeo, *Analytical Chemistry*, Vol 20, 1948, pp. 1014–19. At the present time, the only manufacturing source is the Rankin Glassblowing Co., 3990 Franklin Canyon Road, Martinez, Calif.

It was the original intention of Section E, Research Division VIII, of Committee D-2, in developing this proposed method to use a thermometer as the vapor temperature measuring device. A considerable amount of the early cooperative laboratory work was carried out using the A.S.T.M. thermometer 3 F - 49, but the reproducibility obtained was not satisfactory. Later experience with the special thermocouple, now described in the method, gave improved reproducibility, and it was therefore adopted as the sole means of vapor temperature measurement for continuation of the work of development and for experiments intended to determine correlations for vapor temperature with pressure.

At the time of publication of this method for information, efforts to specify a thermometer as an alternate means of measuring vapor temperatures in this method have not been successful since the differences in readings obtained by the thermocouple and available thermometers appear to vary with the boiling range of the sample being distilled. Further study of this problem may lead to specifications for a thermocouple and thermometer which can be used interchangeably.

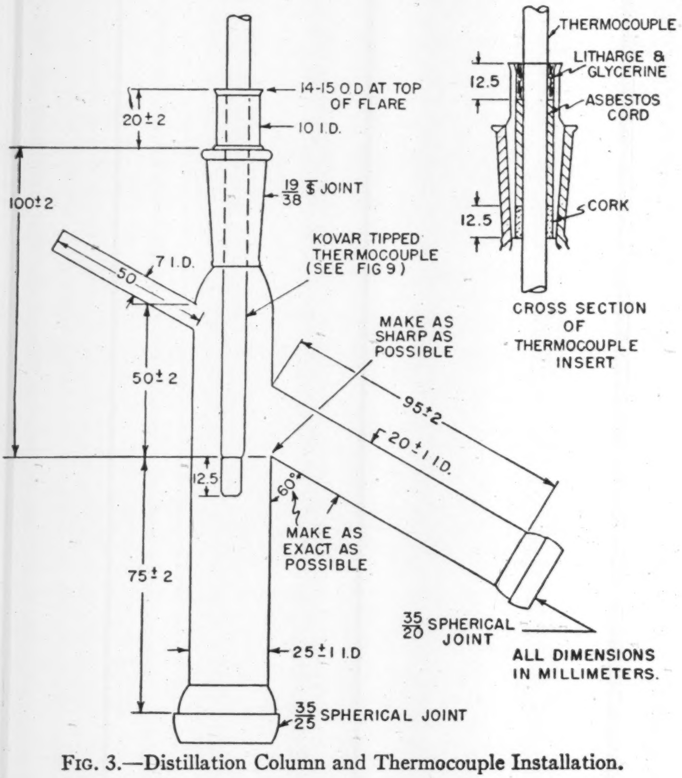

14-15 O D AT TOP OF FLARE
10 I.D.
20±2
100±2
19/38 ₮ JOINT
KOVAR TIPPED THERMOCOUPLE (SEE FIG 9)
7 I.D.
50
50±2
MAKE AS SHARP AS POSSIBLE
95±2
20±1 I.D.
12.5
60°
MAKE AS EXACT AS POSSIBLE
75±2
35/20 SPHERICAL JOINT
25±1 I.D
35/25 SPHERICAL JOINT
ALL DIMENSIONS IN MILLIMETERS.
THERMOCOUPLE
LITHARGE & GLYCERINE
12.5
ASBESTOS CORD
CORK
12.5
CROSS SECTION OF THERMOCOUPLE INSERT

FIG. 3.—Distillation Column and Thermocouple Installation.

NOTE 11.—If a solenoid valve or other electrically operated regulator is used, a suitable manostat is required for activation of the regulating device. Many such manostats are described in the literature or are supplied by laboratory warehouses. As an alternate for the separate manostat and solenoid, a "Cartesian Manostat"[10] may be used. This device is capable of maintaining the system pressure within the specified limits down to a pressure of about 10 mm. of mercury. Also available commercially is a "package" manostat assembly[11] consisting of an air leak, controlled by a diaphragm-type pressure indicator together with all the necessary electrical equipment, valves, etc. This "package" is used without an additional solenoid or regulator.

Sample

A5. *Dehydration of Sample.*—The following is

---

[10] Available from the Emil Greiner Co., 20-26 N. Moore St., New York 13, N. Y.

[11] Available from Precision Scientific Co., 3737 W. Cortland St., Chicago 47, Ill.

suggested as a convenient means of dehydrating samples to be subjected to this distillation test. Heat 300 ml. of the sample to 180 F. (82 C.) and stir vigorously for 1t to 15 min. after adding 10 to 15 g. of 8 to 12-mesh calcium chloride. Allow the mixture to cool without stirring, and remove the oil laryer by decantation.

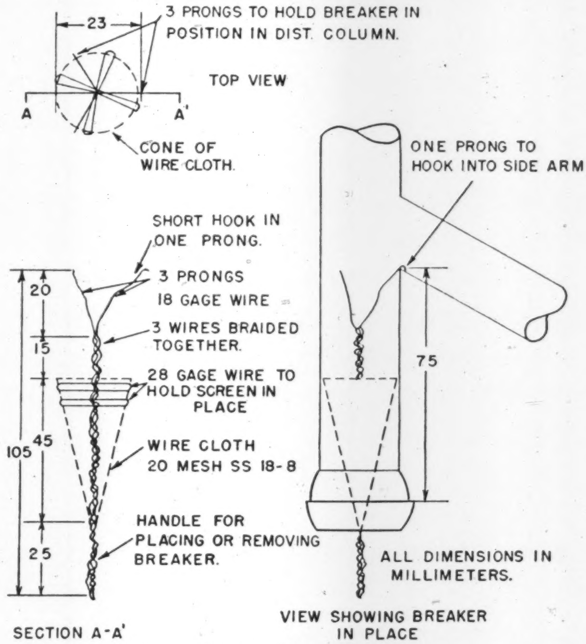

23
3 PRONGS TO HOLD BREAKER IN POSITION IN DIST. COLUMN.
TOP VIEW
A
A'
CONE OF WIRE CLOTH.
ONE PRONG TO HOOK INTO SIDE ARM
SHORT HOOK IN ONE PRONG.
3 PRONGS 18 GAGE WIRE
3 WIRES BRAIDED TOGETHER.
28 GAGE WIRE TO HOLD SCREEN IN PLACE
WIRE CLOTH 20 MESH SS 18-8
HANDLE FOR PLACING OR REMOVING BREAKER.
20
15
45
105
25
75
ALL DIMENSIONS IN MILLIMETERS.
SECTION A-A'
VIEW SHOWING BREAKER IN PLACE

FIG. 4.—Foam Breaker.

Suppression of Foaming of the Sample

A6. The tendency of samples to "bump" or foam excessively is frequently a serious obstacle to the successful distillation of petroleum products under vacuum. In some cases, this is due to the presence of water or dissolved gases, but many samples foam even when apparently free from these contaminants. There is no unanimity of opinion concerning the best way to reduce excessive foaming to manageable proportions. The methods listed below are offered solely as examples of means that have been employed successfully for that purpose.

(*a*) *Dehydration.*—This is referred to in the immediately preceding Section A5.

(*b*) *Degassing.*—The procedure described in Section 7(*e*) is intended to promote degassing. **Slow rates of pressure reduction and of temperature increase for the oil in the flask are important factors in achieving success by this means. Another method of degassing is to filter the sample under vacuum before weighing.**

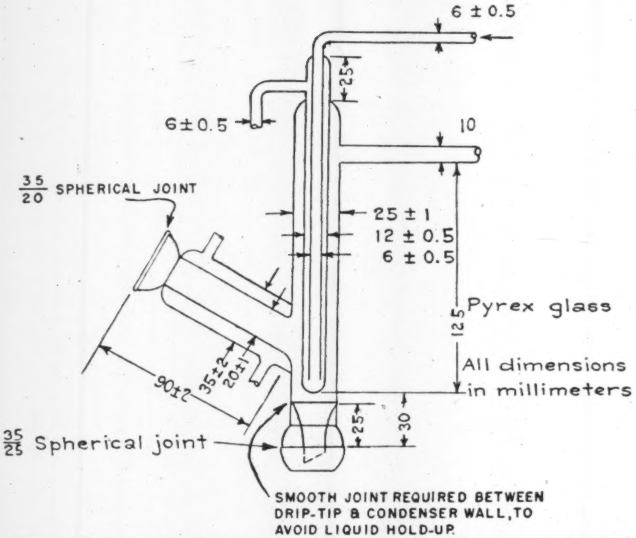
(*c*) *Application of Steel Wool:*

(*1*) **Take about 10 g. of a folded pad of medium grade steel wool. Unfold, and separate into 8 to 10 long loose strands. Push each strand** separately into the bulb of the flask. Avoid packing tightly or the formation of large void spaces. Fill the upper half of the bulb with steel wool, but do not allow any strand to protrude more than ¼ in. into the neck of the flask.

Or, (*2*) Take 0.5 to 0.6 g. of grade 2 steel wool. Roll into five balls, each approximately 8 to 10 mm. in diameter and drop into the flask.

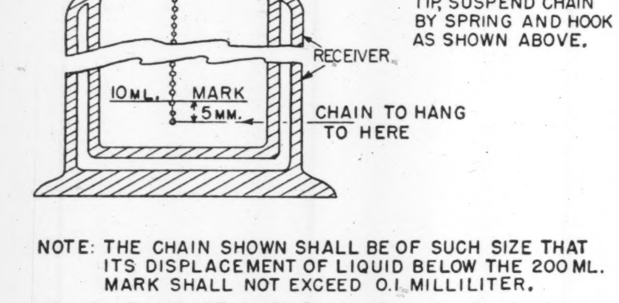
(*d*) *Boiling Chips.*—These consist of broken pieces of porcelain drying plates or broken alundum thimbles which are dropped into the flask before starting a distillation. Hengar granules of the plain type, as used in Kjeldahl nitrogen determinations, are also used in the same way.

(*e*) *Silicone Fluids.*—The addition of one or two drops of Dow Corning Silicone Fluid No. 200[4] (350 centistokes) to the sample in the flask


6 ± 0.5
25
6±0.5
10
$\frac{35}{20}$ SPHERICAL JOINT
25 ± 1
12 ± 0.5
6 ± 0.5
125
Pyrex glass
All dimensions in millimeters
90±2
35±2
20±1
$\frac{35}{25}$ Spherical joint
25
30
SMOOTH JOINT REQUIRED BETWEEN DRIP-TIP & CONDENSER WALL, TO AVOID LIQUID HOLD-UP.

NOTE.—Glassware fabricators have advised that the sidearm condenser jacket cannot conveniently be sealed to the vertical condenser as shown. Accordingly, the length (90 ± 2 mm.) of the condenser and joint is changed to 135 ± 5 mm. to permit ring-seal joining of the jacket to the inner tube in conformity with standard practice. The angle of the junction of the sidearm condenser with the vertical condenser shall be 60 ± 2 deg.

FIG. 5.—Condenser.


CONDENSER DRIP-TIP
WIRE HOOK 30 GAGE OR FINER
SMALL HOLE FOR HANGING WIRE
SPRING
HOOK
IF NO SMALL HOLE IS AVAILABLE IN CONDENSER DRIP-TIP, SUSPEND CHAIN BY SPRING AND HOOK AS SHOWN ABOVE.
RECEIVER
10 ML. MARK
5 MM.
CHAIN TO HANG TO HERE

NOTE: THE CHAIN SHOWN SHALL BE OF SUCH SIZE THAT ITS DISPLACEMENT OF LIQUID BELOW THE 200 ML. MARK SHALL NOT EXCEED 0.1 MILLILITER.

FIG. 6.—Detail of Drip-Chain Attachment to Condenser.

is effective in the suppression of foam in many cases.

(*f*) *Flask Preparation.*—Some laboratories have treated the inside of the flask, prior to use for distillation, in order to provide an active ebullition surface. Methods used for this purpose include: boiling 100 ml. of 33 per cent sodium hydroxide solution for 15 to 20 min., etching of the inside of the flask bottom with hydrofluoric acid fumes, the fusion of fine carborundum or fritted glass to the inside of the flask bottom.

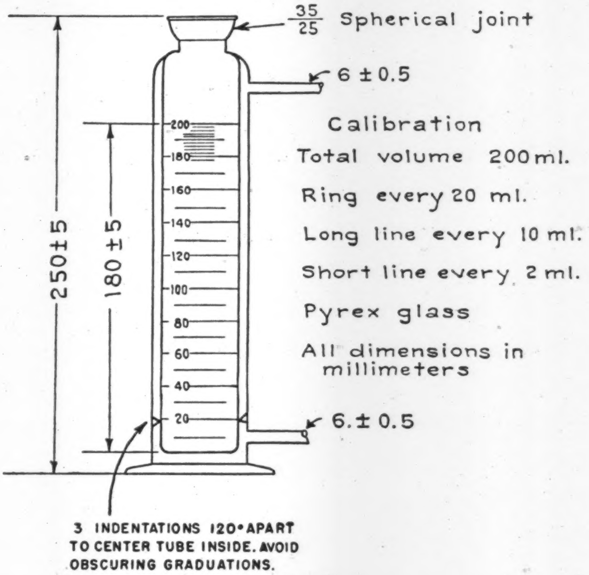

$\frac{35}{25}$ Spherical joint
6 ± 0.5
Calibration
Total volume 200 ml.
Ring every 20 ml.
Long line every 10 ml.
Short line every 2 ml.
Pyrex glass
All dimensions in millimeters
6. ± 0.5
250±5
180 ±5
200
180
160
140
120
100
80
60
40
20
3 INDENTATIONS 120° APART TO CENTER TUBE INSIDE. AVOID OBSCURING GRADUATIONS.

FIG. 7.—Receiver.

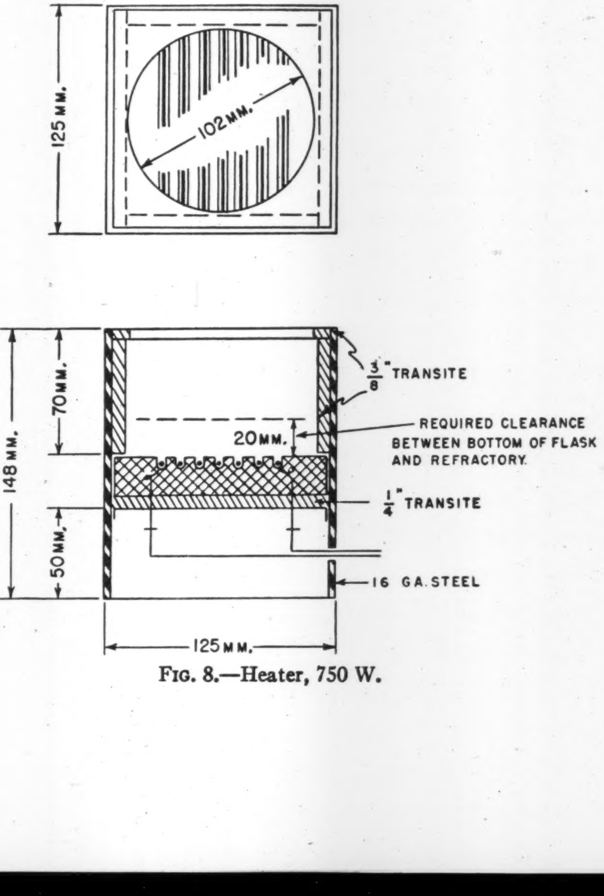

125 MM.
102 MM.
$\frac{3}{8}$" TRANSITE
REQUIRED CLEARANCE BETWEEN BOTTOM OF FLASK AND REFRACTORY.
20 MM.
70 MM.
148 MM.
$\frac{1}{4}$" TRANSITE
50 MM.
16 GA. STEEL
125 MM.

FIG. 8.—Heater, 750 W.

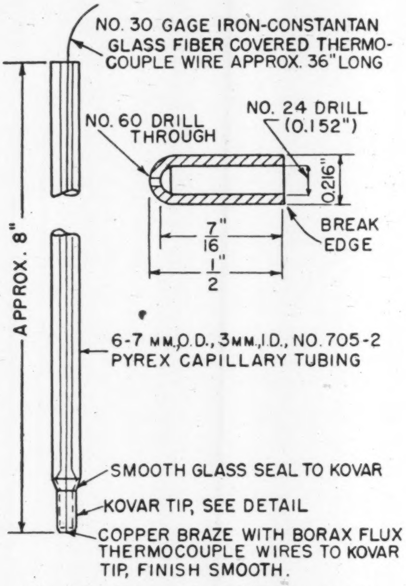

NO. 30 GAGE IRON-CONSTANTAN GLASS FIBER COVERED THERMOCOUPLE WIRE APPROX. 36" LONG
NO. 24 DRILL (0.152")
NO. 60 DRILL THROUGH
0.216"
BREAK EDGE
7"/16
1"/2
APPROX. 8"
6-7 MM.O.D., 3MM.,I.D., NO. 705-2 PYREX CAPILLARY TUBING
SMOOTH GLASS SEAL TO KOVAR
KOVAR TIP, SEE DETAIL
COPPER BRAZE WITH BORAX FLUX THERMOCOUPLE WIRES TO KOVAR TIP, FINISH SMOOTH.

FIG. 9.—Kovar Tipped Thermocouple.

APPENDIX VIII

PROPOSED DEFINITION AND SPECIFICATIONS FOR TRACTOR FUELS[1]

These proposed definitions and specifications are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Definition

1. *Farm Tractor Fuel* means any petroleum product, exclusive of gasoline, diesel fuel and liquefied petroleum gases, which is used for the generation of power for the operation of farm implements and conforms to the specifications for one of the grades prescribed in Section 2.

Specifications

2. The two grades of tractor fuel shall conform to the following requirements:

LIGHT GRADE

A.S.T.M. Distillation:
10 per cent Point, Recovered....347 F., max.
95 per cent Point, Recovered....465 to 518 F.
Octane No., A.S.T.M. Motor Method...35, min.
Corrosion, Copper Strip Test, 3 hr. at 122 F.
......pass. Sulfur......1 per cent, max.

REGULAR GRADE

A.S.T.M. Distillation:
10 per cent Point, Recovered....347 to 401 F.
95 per cent Point, Recovered....465 to 518 F.
Octane No., A.S.T.M. Motor Method...35, min.
Corrosion, Copper Strip Test, 3 hr. at 122 F.
......pass. Sulfur......1 per cent, max.

Methods of Testing

3. The requirements enumerated in Section 2 shall be determined in accordance with the following methods of testing of the American Society for Testing Materials:

(*a*) *Distillation.*—Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86).[2]

(*b*) *Octane Number.*—Standard Method of Test for Knock Characteristics of Motor Fuels by the Motor Method (A.S.T.M. Designation: D 357).[2]

(*c*) *Corrosion.*—Tentative Method of Test for Free and Corrosive Sulfur in Petroleum Products (A.S.T.M. Designation: D 130).[3]

(*d*) *Sulfur.*—Tentative Method of Test for Sulfur in Petroleum Products by the Lamp—Gravimetric Method (A.S.T.M. Designation: D 90).[3]

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[1] **This proposed definition and specification are under the jurisdiction of the A.S.T.M. Committee D-2 on Petroleum Products and Lubricants. Published as information, June, 1950.**

[2] 1949 Book of Standards, Part 5.

[3] 1950 Supplement to Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE D-3
ON
GASEOUS FUELS[1]

The past year was a period of marked activity for Committee D-3 on Gaseous Fuels and its subcommittees. Two new methods, covering methods of test for specific gravity of gaseous fuels and for measurement of gaseous fuel samples, were completed and accepted as tentative. Substantial progress was also made in the preparation of several others.

Meetings of Committee D-3 were held in Atlantic City on June 28, 1949, and in Pittsburgh on February 28, 1950. Several of the subcommittees also met at these times.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. W. Gauger.
Vice-Chairman, E. F. Schmidt.
Secretary, K. R. Knapp.
Advisory Committee: S. W. Burdick, R. L. Dodge, E. O. Mattocks, and Martin Shepherd.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1949 Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of Test for:

Specific Gravity of Gaseous Fuels (D 1070 - 49 T), and Measurement of Gaseous Fuel Samples (D 1071 - 49 T).

These recommendations were accepted by the Standards Committee on September 22, 1949, and the new tentative methods appear in the 1949 Book of A.S.T.M. Standards, Part 5, and in the 1949 Compilation of A.S.T.M. Standards on Gaseous Fuels.

Activities of Subcommittees

Subcommittee I on Collection of Gaseous Samples (E. O. Mattocks, chairman).—A revised draft of a proposed tentative method for sampling natural gas was prepared and distributed to the subcommittee members for review. This resulted in some final editorial revisions which are now being incorporated, preparatory to submitting the final draft to Committee D-3 for approval.

A revised draft of a proposed tentative method for sampling manufactured gas also was prepared and distributed to the subcommittee. Comments received indicated that further revision would be necessary and a new draft will be prepared.

It is hoped that a method for sampling liquefied petroleum gases will be in readiness for subcommittee distribution in the near future.

Subcommittee II on Measurement of Gaseous Samples (H. S. Bean, chairman).—The new Tentative Methods for Measurement of Gaseous Fuel Samples (D 1071 - 49 T) were prepared by this subcommittee.

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. F. Schmidt, chairman).—As the result of much work and study, including an extensive investigation of contemporary types of specific gravity apparatus, the new Tentative Methods of Test for Specific Gravity of Gaseous Fuels (D 1070 - 49 T) were completed by this subcommittee. Following their acceptance by the Standards Committee, these methods were published in the 1949 compilation of "A.S.T.M. Standards on Gaseous Fuels." The members of Committee D-3 were requested that those familiar with specific gravity testing submit comments on the published tentative method in order that any revision or correction may be made in the next printing.

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.**

Subcommittee V on Determination of Special Constituents of Gaseous Fuels.—At the last meeting of the subcommittee, agreement was reached to write up the referee method for determination of total sulfur, including the scope of this method and its recognized limitations, so that distribution could be made to the members for study and comment. It was also decided to establish a task force to study effects of metering in sulfur removal and the problems involved. Further examination of several methods of sulfur removal was also proposed with the objective of developing one for use with gaseous fuels.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (A. W. Gauger, chairman).—A proposed tentative method for measuring the water vapor content of fuel gases by the dew-point method was distributed to subcommittee members for letter ballot in advance of the February 27, 1950, meeting. Comments received were discussed at this meeting and the method was approved with suitable revisions to cover them. The revised method is now the subject of letter ballot in Committee D-3.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (Martin Shepherd, chairman).—Two proposed methods for analysis have been prepared and presented to Committee D-3 for letter ballot approval. These cover analysis of natural gases by the volumetric-chemical method and analysis of natural gases and related types of gaseous mixtures by the mass spectrometer. Active work is now in progress on the two other proposed methods. A sample is also in preparation for analysis by various methods.

This report has been submitted to letter ballot of the committee which consists of 34 members; 34 members returned their ballots, of whom 32 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

A. W. GAUGER,
Chairman.

K. R. KNAPP,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Test for Analysis of Natural Gases by the Volumetric Chemical Method (D 1136 – 50 T), and
Test for Analysis of Natural Gases and Related Types of Gaseous Mixtures by the Mass Spectrometer (D 1137 – 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new tentative methods appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

On December 14, 1950, the Administrative Committee on Standards accepted the recommendation of Committee D-3 that the Method of Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew Point Temperature be published as tentative. The new tentative method appears in the 1950 Supplement to Book of ASTM Standards, Part 5, bearing the designation D 1142 – 50 T.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS[1]

Committee D-4 on Road and Paving Materials has held three meetings during the past year: in Atlantic City, N. J., on June 29, 1949; in San Francisco, Calif., on October 12, 1949; and in Pittsburgh, Pa., on March 2, 1950.

The committee has lost a number of long-time members by death during the past year. Ernest H. Nichols, who died on June 14, 1949, had been a committee member since 1940. Henry M. Milburn, a member since 1919 and active on many subcommittees, died on September 20, 1949. Charles S. Reeve, who had been a member of Committee D-4 since 1912, was formerly chairman of the major subcommittee responsible under the original organization for all methods and specifications relating to bituminous road and paving materials, and subsequently was third vice-chairman in charge of Group B subcommittees. His sudden death on January 6, 1950, brings a feeling of loss, not only to the members of Committee D-4 but also to many other Society members familiar with his interest in various A.S.T.M. activities. Irvin S. Badger, a member since 1948, and a valuable member of Subcommittee C-6 on Brick and Block Pavements, died on March 4, 1950. In the death of Chester A. Hogentogler on June 18, 1949, the committee lost a former active member whose achievements in developing the science and practice of soils engineering were widely recognized.

In appreciation of the services of these gentlemen to the committee and in recognition of their contributions to our knowledge of highway materials, Committee D-4 has adopted a number of Memorial Resolutions, copies of which have been sent to their families and to the President of the Society.

Committee D-4 has several members whose committee activities extend back to the earlier days of the Society and who have been major contributors to the development of specifications and methods of testing long recognized as standard. Committee D-4 has elected two of these members to honorary membership in the committee: A. W. Dow, president of A. W. Dow, Inc., and a member of Committee D-4 since 1903; and H. H. Scofield, professor of Testing Materials, emeritus, Cornell University, and a member of Committee D-4 since 1914.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-4 presented to the Society, through the Administrative Committee on Standards, recommendations for publication of the following:

Tentative Specifications for:

Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 - 49 T),

Tentative Methods of Test for:

Compressive Strength of Bituminous Mixtures (D 1074 - 49 T), and

Effect of Water on Cohesion of Compacted Bituminous Mixtures (D 1075 - 49 T),

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

Tentative Revisions of Standard Methods of Test for:

Determination of Bitumen (D 4 – 42), and
Penetration of Bituminous Materials (D 5 – 49),

Tentative Revision of Recommended Practice for:

Bituminous Paving Plant Inspection
(D 290 – 39).

The tentative specifications, tentative methods of test, tentative revision of Standard Method D 4, and tentative revision of Recommended Practice D 290 were accepted by the Administrative Committee on Standards on September 22, 1949. The tentative revision of Standard Method D 5 was accepted on January 25, 1950.

TABLE I.—COOPERATIVE TESTS ON BITUMEN CONTENT OF PAVING MIXTURES.

Cooperators	Asphalt Concrete Laboratory Mix (G) Aggregate passing ½ in., per cent Sample A	Asphalt Concrete Laboratory Mix (G) Aggregate passing ½ in., per cent Sample B	Sheet Asphalt Plant Mix (C-1) Aggregate passing ¼ in., per cent Sample A	Sheet Asphalt Plant Mix (C-1) Aggregate passing ¼ in., per cent Sample B	Sheet Asphalt Pavement (C-2) Aggregate passing ¼ in., per cent Sample A	Sheet Asphalt Pavement (C-2) Aggregate passing ¼ in., per cent Sample B	Tar Concrete Laboratory Mix (H-1) Aggregate passing ½ in., per cent Sample A	Tar Concrete Laboratory Mix (H-1) Aggregate passing ½ in., per cent Sample B	Tar Concrete Laboratory Mix (H-2) Aggregate passing ½ in., per cent Sample A	Tar Concrete Laboratory Mix (H-2) Aggregate passing ½ in., per cent Sample B
A	6.55	6.50	7.96	7.93	7.29	7.17	6.42	6.94	6.79	6.98
B	6.28	6.34	7.77	7.77	....	....	6.11	6.13	....	....
C	6.39	6.47	7.81	7.85	7.16	7.03	6.53	5.65	6.11	6.17
D	6.30	6.30	7.90	7.80	7.00	7.10	5.80	5.90	6.52	6.51
E	6.46	6.39	7.74	7.64	6.98	6.98	6.20	6.09	6.46	5.79
F	6.30	6.25	7.80	7.80	7.30	7.1	6.0	6.1	6.0	5.9
G	6.42	6.26	7.75	7.65	6.93	6.86	6.12	5.92	6.34	6.25
H	6.42	6.26	7.80	7.81	7.52	7.19	6.08	5.91	6.19	6.18
I	6.40	6.40	7.70	7.70	7.15	7.14	5.2	6.0	6.23	6.61
Average	6.39	6.35	7.80	7.77	7.17	7.07	6.05	6.07	6.33	6.30
Minimum	6.28	6.25	7.70	7.64	6.93	6.86	5.20	5.65	6.00	5.79
Maximum	6.55	6.50	7.96	7.93	7.52	7.19	6.53	6.94	6.79	6.98

On May 8, 1950, the Administrative Committee on Standards accepted the recommendation of Committee D-4 that the Standard Recommended Practice for Bituminous Paving Plant Inspection (D 290 – 39) be withdrawn.

The new Tentative Specifications for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 – 49 T) replace the former Standard Specifications for Sand for Sheet Asphalt and Bituminous Concrete Pavements (D 162 – 29), which were withdrawn in 1949. Terminology and requirements of the former standard have been changed to be consistent with Tentative Specifications for Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surfaces Courses (D 947 – 49 T) and Tentative Specifications for Asphaltic Mixtures for Sheet Asphalt Pavements (D 978 – 49 T), recently approved for publication.

The Tentative Method of Test for Compressive Strength of Bituminous Paving Mixtures is one of several methods which are being standardized by Committee D-4, and is an essential part of the new Tentative Method of Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures. Standardization of these procedures is necessary for their application in research and in practical engineering use. There has long been a demand for a laboratory procedure for determining the effect of water and moisture on the service performance of bituminous paving mixtures.

The tentative revision of Standard Method of Test D 4 – 42 is partly editorial for the purpose of clarifying details of procedure, and also modifies Method No. 2 of the standard to reduce the time required for completing determinations of bitumen in materials containing fine mineral matter.

The tentative revision of Standard

Method of Test D 5 - 49 provides for testing bituminous materials with penetrations of 200 or less in a 3-oz. container, after minimum periods of 1 hr. in air and 1 hr. in a controlled temperature bath, thus restoring these requirements of the former standard, D 5 - 25, as applied to bituminous materials of the lower range in penetration.

The tentative revisions of the Recommended Practice for Bituminous Paving Plant Inspection are mainly editorial in nature, intended to bring the terminology into accord with present practice and to make suitable reference to A.S.T.M. standards and tentatives approved in recent years.

NEW TENTATIVE

The committee recommends the publication of a new Tentative Method of Test for Bitumen Content of Paving Mixtures, as appended hereto.[2] The proposed method is in accordance with practice which has been followed for many years by a large number of laboratories engaged in the testing of asphaltic paving mixtures and should be standardized by the Society. Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures (R. R. Thurston, chairman) conducted cooperative tests in developing the recommended tentative method and results of these determinations, made in duplicate on five samples by nine laboratories, are presented in Table I.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee D-4 recommends that the following revisions be immediately incorporated in the standard, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the recommendation may be referred to letter ballot of the Society.

Standard Specifications for Asphalt Plank (D 517 - 40)[3]

Section 11.—Delete all of Section 11, including the heading "Indentation," and substitute the following:

11. *Hardness.*—The hardness of the plank, when tested in accordance with the method prescribed in Section 17, shall meet the following requirements:

Temperature, deg. Fahr.	Load, lb.	Penetration, in.
115	50	not more than 0.25
77	200	not more than 0.25
39	1000	not less than 0.10

Section 17.—Delete all of Section 17, including the heading "Identation Test," and substitute the following:

17. *Hardness.*—The hardness of the plank shall be determined by means of a penetrometer in which the pin in contact with the plank during the test is a right cylinder ⅝ in. in diameter for a distance of at least 1½ in. from its lower end. The specimen shall be kept submerged in water maintained at the temperature specified for a time sufficient to insure that the specimen has attained this temperature throughout. The test shall be made with the specimen submerged in water at the temperatures indicated in Section 11 and supported on a smooth, solid support not less than 2 in. in diameter and concentric with the contact pin of the penetrometer. The tests shall be performed upon samples at least 6 in. in length and of the full width of the plank. In each case the required load shall be applied for a period of 60 sec., and the contact pin of the penetrometer shall be applied to the specimen at points 1½ in. or more from the edge of the specimen.

TENTATIVES CONTINUED WITHOUT REVISION

The subcommittees of Committee D-4 and the joint subcommittee of Committees D-4 and D-18 have reviewed existing tentative specifications and methods of test which have been published by the Society for two years or more without revision, and Committee

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[2] This method was accepted as tentative by the Society and appears in the 1950 Supplement to ASTM Book of Standards, Part 3.

[3] 1949 Book of A.S.T.M. Standards, Part 3.

D-4 recommends that the following be continued as tentative without revision:

Tentative Specifications for:

Materials for Stabilized Base Course (D 556 - 40 T)[3] and Materials for Stabilized Surface Course (D 557 - 40 T).[3]—The joint subcommittee of Committees D-4 and D-18 is cooperating with a committee of the American Association of State Highway Officials in an effort to combine these two specifications and to prepare simplified and more direct requirements for suitable materials.

Asphalt Cements for Use in Pavement Construction (D 946 - 47 T).[3]—Subcommittee C-1 is engaged in a study of additional requirements involving improved control of the properties and uniformity of asphalt cements.

Preformed Expansion Joint Filler for Concrete (Bituminous Type) (D 994 - 48 T).[3]—This pecification should have further consideration by Subcommittee D-3, which has been inactive during the past year because of the difficulty of replacing the former chairman.

Tentative Methods of Test for:

Loss on Heating of Oil and Asphaltic Compounds (D 6 - 39 T).[3]—This method of test has been in extensive use for many years, and should be retained until an improved procedure can be adopted. A subcommittee is actively engaged in studying additional limitations for apparatus used in the method and the possibilities of developing an improved procedure.

Moisture-Density Relations of Soils (D 698 - 42 T).[3]—This method is under the joint jurisdiction of Committee D-4 and Committee D-18 on Soils for Engineering Purposes. In its report at the 1949 Annual Meeting, Committee D-4 recommended that this method be approved for reference to letter ballot of the Society for adoption as standard. This year the recommendation was continued. However, Committee D-18 recommended that the method be continued as tentative without revision.

Soil Bituminous Mixtures (D 915 - 47 Г).[3]—A subcommittee of Committee D-18 is planning to study this procedure further in correlation with construction control and service performance of stabilized highway soils. Such an investigation should be made to demonstrate the applicability and utility of the tentative method under practical conditions and is desirable as a basis for further action by Committees D-4 and D-18.

Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (D 916 - 47 T).[3]—The study of this method by use in field projects, mentioned in the 1949 report of Committee D-4, has not been completed. It is expected that extensive use of the method by the committee of the Highway Research Board may lead to improvements in the apparatus or testing procedure.

The recommendations appearing in this report have been approved by letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) has prepared a proposed tentative method of test for the resistance to plastic flow of fine-aggregate bituminous mixtures. This method of test is commonly known as the Hubbard-Field Stability Test and is widely used for the evaluation of paving mixtures, comprising aggregates of small size. The subcommittee is also considering a method for the determination of specific gravity of compressed bituminous mixtures. The Triaxial Institute, which is functioning as a project committee of Subcommittee B-2, is engaged in a comprehensive research program involving development of apparatus and testing procedures applicable to bituminous mixtures and correlation of triaxial test results with service performance and other methods for physical testing. This subject was an important topic of discussion at the San Francisco meetings of the Society and of Committee D-4.

Subcommittee B-5 on Softening Point (I. E. Manning, chairman) is planning cooperative testing to study several features of the ring-and-ball softening point tests in an effort to develop a

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

TABLE II.—RESULTS OF COOPERATIVE TESTS FOR DETERMINATION OF BITUMEN, SAMPLE OF ASPHALT CEMENT MIXED WITH POWDERED FILLER.

Co-operator	Bitumen (Soluble in CS_2), per cent		Mineral Matter (Ash), per cent		Difference (Organic Insoluble), per cent	
	Determinations	Average	Determinations	Average	Determinations	Average
A........	81.12		17.32		1.56	
	81.09	81.13	17.39	17.36	1.52	1.51
	81.18		17.36		1.46	
B........	80.5	80.7	17.8	17.6	1.7	1.7
	80.8		17.5		1.7	
C........	81.09		17.32		1.59	
	81.23	81.18	17.32	17.30	1.45	1.52
	81.22		17.26		1.52	
D........	80.93		17.07		2.00	
	80.99	81.00	17.00	16.99	2.01	2.01
	81.08		16.89		2.03	
E........	81.11		17.31		1.58	
	81.09	81.09	17.35	17.33	1.56	1.57
	81.08		17.34		1.58	
F........	80.95		17.78		1.27	
	81.05	81.00	17.65	17.68	1.30	1.32
	81.00		17.60		1.40	
G........	81.08	81.05	17.48	17.47	1.44	1.48
	81.01		17.46		1.53	
H........	81.16		17.40		1.44	
	81.21	81.19	17.54	17.46	1.25	1.35
	81.20		17.45		1.35	
I........	81.02		17.21		1.77	
	80.90	80.95	17.25	17.23	1.85	1.82
	80.92		17.23		1.85	
J........	81.04		17.18		1.78	
	80.79	80.85	17.43	17.37	1.78	1.78
	80.71		17.51		1.78	

TABLE III.—RESULTS OF COOPERATIVE TESTS FOR DETERMINATION OF BITUMEN, SAMPLE OF REFINED TRINIDAD ASPHALT.

Co-operator	Bitumen (Soluble in CS_2), per cent		Mineral Matter (Ash), per cent		Difference (Organic Insoluble), per cent	
	Determination	Average	Determination	Average	Determination	Average
A........	55.5		36.4		8.1	
	55.6	55.4	36.4	36.5	8.0	8.1
	55.1		36.6		8.3	
D........	56.5		34.2		9.3	
	56.9	56.7	33.9	34.1	9.2	9.2
E........	55.4		36.5		8.1	
	55.5	55.5	36.4	36.4	8.1	8.1
F........	56.1		35.8	35.7	8.1	
	56.1	56.1	35.6		8.3	8.2
G[a].......	55.7		36.4		7.9	
	55.8	55.8	36.1	36.2	8.1	8.0
K........	53.39	54.91	35.97		10.64	
	56.06		34.87	35.36	9.07	9.73
	55.27		35.24		9.49	

[a] Weight of Sample, 0.5 g.; weight of asbestos mat, 0.1 g.

TABLE IV.—RESULTS OF COOPERATIVE TESTS FOR DETERMINATION OF BITUMEN, SAMPLE OF TRINIDAD ASPHALT CEMENT, 62 PENETRATION.

Co-operator	Bitumen (Soluble in CS_2), per cent		Mineral Matter (Ash), per cent		Difference (Organic Insoluble), per cent	
	Determinations	Average	Determinations	Average	Determinations	Average
A........	71.4		23.7		4.9	
	71.2	71.2	23.6	23.7	5.2	5.1
	71.0		23.8		5.2	
D........	72.5		19.1	19.5	8.4	
	72.2	72.3	19.8		8.0	8.2
E........	71.3		23.4		5.0	
	71.1	71.2	23.5	23.5	5.1	5.1
F........	74.6		20.2		5.2	
	74.5	74.6	20.6	20.4	4.9	5.0
G[a].......	71.2		23.1		5.7	
	71.3	71.3	23.2	23.1	5.5	5.6
K........	72.68		22.16		5.16	
	70.67	71.40	23.68	22.92	5.65	5.68
	70.86		22.91		6.23	

[a] Weight of sample, 1.0 g. and 0.5 g., respectively; weight of asbestos mat, 0.1 g.

TABLE V.—RESULTS OF COOPERATIVE TESTS FOR DETERMINATION OF BITUMEN, SAMPLE OF TRINIDAD ASPHALT CEMENT, 104 PENETRATION.

Co-operator	Bitumen (Soluble in CS_2), per cent		Mineral Matter (Ash), per cent		Difference (Organic Insoluble), per cent	
	Determinations	Average	Determinations	Average	Determinations	Average
A........	75.3		20.4		4.3	
	75.2	75.3	20.3	20.3	4.5	4.4
	75.5		20.1		4.4	
D........	75.4		19.5		5.1	
	75.8	75.6	18.8	19.2	5.4	5.3
E........	75.3		20.2		4.5	4.5
	75.4	75.4	20.1	20.2	4.5	
F........	77.2		18.7		4.1	
	77.0	77.1	19.1	18.9	3.9	4.0
G[a].......	75.1	75.1	19.8	19.7	5.1	5.2
	75.1		19.6		5.3	
K........	75.29		20.08		4.63	
	75.23	75.34	19.67	19.95	5.10	4.71
	75.5		20.1		4.4	

[a] Weight of sample, 0.5 g; weight of asbestos mat, 0.1 g.

satisfactory method combining two methods now published by the Society.

Subcommittee B-7 on Viscosity and Float Tests (C. A. Benning, chairman) has a proposed method for the determination of Engler specific viscosity in preparation, which will probably be recommended to the committee for approval during the coming year.

Subcommittee B-21 on Solubility Tests (E. H. Porter, chairman) conducted a series of cooperative tests on the determination of bitumen in connection with the tentative revision of Standard Method D 4. A sample of asphalt containing mineral filler and three samples of Trinidad asphalt were used in these tests. The results obtained by a number of laboratories are presented in Tables II, III, IV, and V. This subcommittee is preparing a proposed standard procedure for the direct determination of ash in bituminous materials.

Subcommittee C-4 on Specifications for Emulsified Asphalts (F. H. Baumann, chairman) is considering requirements for an additional grade of emulsified asphalt suitable for surface treatment and having rapid-setting, high-viscosity properties.

Subcommittee C-12 on Bituminous Surface Treatment (W. H. Mills, chairman) has prepared a specification for crushed stone, crushed slag, and crushed gravel for use in single bituminous surface treatments and is considering requirements applicable to other treatments.

There is a need for specifications covering hydrated lime for highway purposes, and Committee D-4 has requested Committee C-7 on Lime to undertake the preparation of specifications for this material.

RESEARCH ACTIVITIES

In its report presented at the 1949 Annual Meeting, Committee D-4 listed a number of subjects on which considerable research is necessary for the development of information essential to the development of reliable methods and specifications for the control of road materials. The subcommittees concerned with these problems are making considerable progress, as evidenced by the acceptance of several new tentative methods during the past year and by the preceding summary of activities for a few subcommittees. Of particular interest is the development of laboratory methods for the physical testing of compressed bituminous mixtures and study of the effects of moisture on their performance. An extensive program is under way, under the direction of Subcommittee B-25, to evaluate the reproducibility of the Tentative Method of Test for Effect of Water on Cohesion of Compacted Bituminous Mixtures. Subcommittee B-26 is studying a difficult problem, that of determining the effect of water on bituminous coatings applied to aggregates. Personal factors in existing methods, involving an evaluation of uncoated areas on aggregates, lead to poor reproducibility, and the subcommittee is attempting to minimize the influence of these factors, studying a variety of suggested procedures.

Present methods for the determination of asphalt content of asphaltic road materials have questionable significance and reliability. Subcommittee B-1 has conducted a number of cooperative test programs, and the results of these tests have indicated that a method involving vacuum or steam distillation may provide adequate information. Further work is in progress.

In the field of aggregates, Subcommittee B-12 is continuing its study of the quality of particles according to hardness and is currently investigating the development of a method for abrasion of sand. Subcommittee B-14 is endeavoring to develop a method for the determination of specific gravity which will be significant in the control and.design of paving mixtures.

Subcommittee B-16 is engaged in a study of possible methods for the evaluation of setting or curing properties of bituminous highway materials, for which there is no generally accepted procedure of demonstrated value at present.

It has been realized for many years that the present standard method for loss on heating of asphaltic road materials is poorly reproducible and that the percentages of volatile materials determined by this method may have little meaning in relation to performance qualities. Subcommittee B-18 is studying apparatus and procedures in the development of an improved method.

Last year, on the initiative of Committees D-4 and D-8, a very successful symposium on accelerated tests for durability of bituminous materials was held during the Annual Meeting. One reason for proposing this symposium was to promote further thought and effort on the problem of adequate methods for evaluating service performance of bituminous materials. Subcommittee B-19 now is considering the development of a program aimed to obtain reliable fundamental information.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, C. E. Proudley.
First Vice-Chairman, Bailey Tremper.
Second Vice-Chariman, A. T. Goldbeck.
Third Vice-Chairman, E. W. Klinger.
Secretary, B. A. Anderton.

This report has been submitted to letter ballot of the committee, which consists of 140 members; 107 members returned their ballots, of whom 100 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. H. BAUMANN,
Chairman.

B. A. ANDERTON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Test for Resistance to Plastic Flow of Fine-Aggregate Bituminous Mixtures (D 1138 - 50 T).

New Tentative Specifications for:

Crushed Stone, Crushed Slag, and Gravel for Single Bituminous Surface Treatment (D 1139 - 50 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE[1]

Committee D-5 on Coal and Coke held meetings in Atlantic City, N. J., on June 29, 1949, and in Pittsburgh, Pa., on February 28, 1950.

During the year one member was added to the committee and one member resigned, resulting in a present total membership of 58, of whom 17 are classified as consumers, 16 as producers, and 25 as general interest members.

Subcommittee XVI on Ignitibility of Coal and Coke (R. A. Sherman, chairman) was discharged because it became evident that further work toward a standard on this subject was not justified unless, and until, there may be developed a more consistent understanding of the meaning of the term "ignitibility" and of the significance of such property in the use of solid fuels.

Chairman W. A. Selvig was a delegate to the Working Party on Classification, Coal Committee, E. C. E., Geneva, Switzerland. He attended two meetings held in Geneva during the year. Since there exists considerable common interest between this group and Technical Committee 27 on Solid Mineral Fuels of the International Organization for Standardization (ISO), such participation by the chairman constitutes liaison with the latter organization, supplementing that which it has been possible to establish by mail. In the absence of direct participation by any Commitmittee D-5 representative in the affairs of ISO (a restriction so far imposed by the cost and time implied in such participation), such liaison has real value.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

In order to promote uniformity in sieve size designations for all tests involving the sieve analysis of coke, Committee D-5 recommends for immediate adoption the following revisions in two standards. The committee requests the necessary nine-tenths affirmative vote at the Annual Meeting so that these recommendations may be submitted to letter ballot of the Society.

Standard Method of Tumbler Test for Coke (**D 294 – 29**):[2]

Section 2 (*b*), *Sieves.*—Change the second sentence now reading "For sieving the coke after the tumbler test, square-mesh sieves having 2, 1.5, 1.06, 0.530, and 0.265-in. actual openings between the wires shall be used" to read "For sieving the coke after the tumbler test, square-mesh sieves having 2, $1\frac{1}{2}$, 1, $\frac{1}{2}$, and $\frac{1}{4}$-in. actual openings between the wires shall be used."

New footnote 3.—Add a footnote reference to Section 2 (*b*) on Sieves, renumbering subsequent footnotes accordingly, and add new footnote 3 which reads "User's attention is called to the fact that yields of certain sizes as determined by use of the specified screens cannot be compared directly with results obtained by using screens as specified prior to the 1950 revision. An important example occurs in the case of the tumbler test stability factor (per cent remaining on 1 in. after tumbling) which will be slightly higher than comparable previous results using the formerly specified 1.06-in. screen."

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 5.

Section 5.—Change the third sentence now reading "All of the coke shall then be removed from the drum and sieved, using the following square-mesh sieves: 2-in., 1.5-in., 1.06-in., 0.530-in., and 0.265-in." to read "All of the coke shall then be removed from the drum and sieved, using the following square-mesh sieves: 2, 1½, 1, ½, and ¼-in."

Section 6.—Change the designations "1.5-in. sieve, 1.06-in. sieve, 0.530-in. sieve, and 0.265-in. sieve" to read "1½-in. sieve, 1-in. sieve, ½-in sieve, and ¼-in. sieve," respectively.

Standard Method of Test for Sieve Analysis of Coke (**D 293 – 29**):[2]

Section 2 (a).—In the table of size *versus* sieve opening, change the lines, "0.265 in. . . . . 0.265", "0.530 in. . . . . 0.530", and "1.06 in. . . . . 1.06" to read "¼ in. . . . . 0.250", "½ in. . . . . 0.500", and "1 in. . . . . 1.00," respectively.

New footnote 3.—Insert a footnote 3 reference after "sieve openings" in Section 2 (*a*) for a new footnote to read: "User's attention is called to the fact that yields of certain sizes as determined by use of the specified screens cannot be compared directly with results obtained by using screens as specified prior to the 1950 revision." Renumber subsequent footnotes accordingly.

Section 4.—Change the third and fourth sentences now reading "For coke 1 in. and larger in size the following square-mesh sieves are usually used: 4.00, 3.00, 2.00, 1.50, 1.06, and 0.530 in. For coke smaller than 1 in. in size the square-mesh sieves usually used are: 0.750, 0.530, 0.375, 0.265, 0.187, and 0.132 in." to read "For coke 1 in. and larger in size the following square-mesh sieves are usually used: 4, 3, 2, 1½, 1, and ½ in. For coke smaller than 1 in. in size the square-mesh sieves usually used are ¾, ½, ⅜, ¼, 0.187, and 0.132 in."

Section 5 (a).—Revise the present report form which reads as follows:

Passing	Retained on	Per cent
	4.00-in. sieve..................	.....
4.00-in. sieve	3.00-in. sieve..................	.....
3.00-in. sieve	2.00-in. sieve..................	.....
2.00-in. sieve	1.50-in. sieve..................	.....
1.50-in. sieve	1.06-in. sieve..................	.....
....	....	.....
....	....	.....
0.087-in. sieve	0.132-in. sieve..................	.....
0.132-in. sieve......	....	.....
Total..................	....	100.0

to read:

Passing	Retained on	Per cent
	4-in. sieve..................	.....
4-in. sieve	3-in. sieve..................	.....
3-in. sieve	2-in. sieve..................	.....
2-in. sieve	1½-in. sieve..................	.....
1½-in. sieve	1-in. sieve..................	.....
....	....	.....
....	....	.....
0.087-in. sieve	0.132-in. sieve..................	.....
0.132-in. sieve......	....	.....
Total..................	....	100.0

TENTATIVES CONTINUED WITHOUT REVISION

Concerning two tentative tests for grindability of coal, namely, Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (D 408 – 37 T) and Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 – 37 T), action taken at the meeting of Committee D-5 in Pittsburgh on February 28, 1950, insures that specific recommendations covering these two tentatives will be made not later than the date of the Society's 1951 Annual Meeting. It is recommended that in the interim they be continued as tentatives.

It is recommended that the Tentative Method of Sampling and Analysis of Coal for Volatile Matter Determination in Connection with Smoke Ordinances (D 980 – 48 T) be continued without change during the coming year, since it is felt that insufficient experience has been accumulated to warrant other action at this time.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee XIII on Coal Sampling (H. F. Hebley, chairman) met in Chicago on March 21, 1950, to discuss the possibility of working toward a standard for the automatic sampling of coal for large users, for use by utilities and coke plants, for example. An interested group from the utilities has advanced both a request for such standardization and an offer of help in the form of a comprehensive experimental program. A special section of the subcommittee, which will include several consulting members from the interested industries, is being constituted to work on this project.

Subcommittee XV on Plasticity and Swelling of Coal (C. C. Russell, chairman) held no meeting during the year but reports considerable progress in the cooperative testing program designed to check the reproducibility of test results by the Gieseler method. Fluidities of samples of flat pitch and road tar have been determined by 16 cooperating laboratories, with another recently enlisted. Variability of results has led to suspicion regarding the advisability of reheating pitch samples, so that two silicone samples are being circulated for additional checks. A new procedure for evaluating friction has been developed, and a new method for calibrating the Gieseler apparatus is under preparation.

Subcommittee XXI on Methods of Analysis (O. W. Rees, chairman) held two meetings during the year. Possible interference effects of oxides of nitrogen in the carbon and hydrogen determinations of the ultimate analysis have been of principle consideration. The matter was resolved with the help of data from five cooperating laboratories, and final methods for these two elements are in preparation. The subcommittee is currently at work on tentative methods for determining nitrogen and mineral CO_2.

This report has been submitted to letter ballot of the committee, which consists of 58 members; 48 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. A. SELVIG,
Chairman.

C. H. SAWYER,
Secretary.

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[3] The letter ballot on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS[1]

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 30, 1949, at Atlantic City, N. J., and on February 24, 1950, at New York, N. Y.

The Advisory Subcommittee held three meetings during the year: on June 29, 1949, at Atlantic City, N. J., on December 2, 1949, and February 24, 1950, in New York, N. Y.

Cooperative relations are being continued between Committee D-6 and other A.S.T.M. committees, as well as with other standardizing bodies.

At the present time, Committee D-6 consists of 84 members, of whom 67 are voting members; 30 are classified as producers, 24 as consumers, 24 as general interest, and 6 as consulting members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. R. Willets.
Vice-Chairman, M. A. Krimmel.
Secretary, R. H. Carter.

NEW TENTATIVES

The committee recommends that the following three methods be accepted for publication as tentative, as appended hereto:[2]

Tentative Methods of Test for:

Static Blending Test for Corrugated Paperboard, and Water Soluble Sulfates in Paper and Paperboard.

Tentative Specifications for:

Filter Paper for Use in Chemical Analysis.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following four tentative methods be approved without change for reference to letter ballot of the Society for adoption as standard:

Tentative Methods of Test for:

Paraffin Wax Absorptiveness of Paper (D 983 - 48 T),[3]
Reducible Sulfur in Paper (D 984 - 48 T),[3]
45-deg., 0-deg. Directional Reflectance for Blue Light (Brightness) of Paper (D 985 - 48 T),[3] and
Preparation of a Magnesium Oxide Standard for Spectral Reflectivity (D 986 - 48 T).[3]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following two revised standard methods, as appended hereto:[4]

Standard Methods of Test for:

Basis Weight of Paper and Paper Products (D 646 - 44),[3] and
Hydrogen Ion Concentration (pH) of Paper Extract (D 778 - 46).[3]

REVISION OF TENTATIVES

The committee recommends for approval and continuation as tentative the following revised tentative method, as appended hereto:[5]

Tentative Method of Test for:

Organic Nitrogen in Paper and Paperboard (D 982 - 48 T).[3]

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[2] The methods were accepted as tentative by the Society and appear in the 1950 Supplement to ASTM Book of Standards, Part 4.

[3] 1949 Book of A.S.T.M. Standards, Part 4.

[4] These recommendations were accepted by the Society and the revised methods appear in the 1950 Supplement to Book of ASTM Standards, Part 4.

[5] The revised method was accepted as tentative by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 4.

This method has been revised to include the use of a standard solution of boric acid and to correct the formula for computing test results.

Recommendation is also made for approval of the following revisions in the *Tentative Method of Test for Scuff Resistance of Paperboard* (**D 1029 - 49 T**):[3]

Title.—Change to read "Tentative Method of Test for Peeling Resistance of Paperboard."

Similarly throughout the text change the term "scuff" or "scuffing" to "peeling" or "rubbing" as appropriately noted.

Section 1.—Add the following sentence at the end of the paragraph (before the Note):

> Because there is still much to be learned about peeling resistance and its measurement, the description of "apparatus" in Paragraph 2 has been limited to a type of machine rather than to an exact description of an existing instrument.

Section 2.—Change the eighth and ninth sentences to read as follows by the addition of the italicized words and the omission of those in brackets:

> The [total] distance of travel of the moving plate shall be 6 in. in each direction, *that is a total of 12 in. for one double stroke.* [In order] *The machine shall be constructed so as* to minimize [the] *any* tendency *of the moving plate* to rotate [about the leading edge of the moving plate, the connection between the driving arm and the moving plate should be as close as possible to the plane of the specimen] *or move laterally.*

Section 7.—Add the following sentence at beginning of the paragraph:

> Results of tests made with grain direction parallel to stroke and perpendicular to stroke are to be reported separately.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentative methods which have stood for two years without revision be continued as tentative:

Tentative Methods of Test for:

Puncture and Stiffness of Paperboard, Corrugated and Solid Fiberboard (D 781 - 44 T),
Analytical Filter Papers (D 981 - 48 T),
Stretch of Paper and Paper Products Under Tension (D 987 - 48 T), and
Water Vapor Permeability of Paper, Paperboard and Other Sheet Materials (D 988 - 48 T).

A revised method for puncture is still under investigation. The methods for analytical filter papers are currently being revised to include several methods which will be called for in the proposed specification for filter paper. Possible modification of the method for stretch to make it adaptable to creped paper is under investigation. The method for water vapor permeability is undergoing revision, principally in format, and will be included in an integrated method now being developed by Committee E-1.

The remaining tentative methods under the jurisdiction of Committee D-6, although requiring no revision, are also recommended for continuation as tentative.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Paper Testing Methods (M. S. Kantrowitz, chairman) held three meetings during the year. The subcommittee has been active in developing new methods of test and has also continued its critical study of various methods of test for paper and paper products that have been adopted by other agencies, for example those of the Technical Association of the Pulp and Paper Industry. The subcommittee prepared one of the two new methods of test which are being recommended for publication as tentative, namely, Water Soluble Sulfates in Paper and Paperboard. The four tentative methods being recommended for adoption as standard, the two standard methods for which revision is recommended for immediate adoption, and Tentative Method D 982, for which revision is recommended, are also under the jurisdiction of this subcommittee.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Methods on the following properties are being given consideration by the subcommittee:

Erasability,
Stiffness and softness,
Gloss,
Lint,
Chloride content,
Moisture expansivity,
Pinholes,
Zinc pigments,
Water soluble matter, and
Color fastness to light.

In addition, studies are being made which may lead to proposed revisions in the Standard Methods of Test for Opacity of Paper and Paper Products (D 589 - 44), Bursting Strength of Paper (D 774 - 46) and Time of Penetration by Water of Sized Paper and Paper Products (D 779 - 46) and in the Tentative Methods of Test for Water Vapor Permeability of Paper, Paperboard and Other Sheet Materials (D 988 - 48 T).

Subcommittee II on Significance of Test Methods (L. F. Reid, chairman) held one meeting during the year. Its current major project consists in the revision of the Monograph on Paper and Paperboard, Characteristics, Nomenclature and Significance of Tests.

Subcommittee III on Specifications for Paper (P. F. Wehmer, chairman) held two meetings during the year. The subcommittee prepared the Specifications for Filter Paper for use in Chemical Analysis, which is being recommended for publication. It has also been studying various specifications for paper and paper products that have been adopted by other agencies, such as the Federal Specifications Board.

Subcommittee IV on Container Board (W. B. Lincoln, Jr., chairman) held two meetings during the year and is currently working on the following test procedures:

Ring crush test,
Flat crush test,
Tensile strength test,
Bursting strength test,
Immersion number,
Puncture test, and
Sampling.

The subcommittee prepared one of the two new methods of test that are being recommended for publication as tentative, namely, Static Bending of Corrugated Board. Tentative Method D 1029 - 49 T, for which revision is recommended, is also under the jurisdiction of this subcommittee.

The establishment of Subcommittee V on Research has been authorized. Its organization will take place in the near future.

The present officers were reelected for the ensuing term of two years.

This report has been submitted to letter ballot of the committee, which consists of 67 voting members; 38 members returned their ballots, of whom 34 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. R. WILLETS,
Chairman.

H. A. BIRDSALL,
Secretary.

REPORT OF COMMITTEE D-7
ON
WOOD[1]

Committee D-7 on Wood held meetings at Atlantic City on June 29, 1949, and at Chicago on March 14, 1950. A number of subcommittees met in conjunction with the meetings of the main committee. L. J. Markwardt was reelected chairman for the ensuing two-year term.

Two new subcommittees were organized during the year: Subcommittee XIII on Durability and Exposure with T. R. Truax, as chairman, and subcommittee XIV on Methods of Chemical Analysis with G. J. Ritter as chairman. In Subcommittee VII on Wood Poles and Cross Arms, a new task group on cross arms, under the chairmanship of L. J. Jacobi, was organized. The development of these fields of activities has added a number of new members to the committee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-7 presented to the Society through the Administrative Committee on Standards new Tentative Methods for Establishing Structural Grades of Lumber (D 245 - 49 T), including the withdrawal of Standard Specifications for Structural Wood Joist and Plank, Beams and Stringers, and Posts and Timbers (D 245 - 37).

These recommendations were accepted by the Standards Committee on September 22, 1949, and the new tentative methods appear in the 1949 Book of A.S.T.M. Standards, Part 4.

NEW TENTATIVES

The committee recommends the publication as tentative of the following specifications and methods, as appended hereto:[2]

Tentative Methods of:

Testing the Integrity of Glue Joints in Laminated Wood Products for Exterior Service.

Tentative Method of Tests for:

Ash in Wood,
Water Solubility of Wood,
Ether-Solubility of Wood,
Alcohol-Benzene Solubility of Wood,
One Per Cent Caustic Soda Solubility of Wood,
Holocellulose in Wood,
Lignin in Wood,
Alphacellulose in Cellulosic Materials.

Tentative Methods for the Preparation of:

Extractive-free Wood.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption as standard the revision, as appended hereto,[3] of the Standard Methods of Testing Small Clear Specimens of Timber (D 143 - 49), and accordingly asks for a nine-tenths affirmative vote in order that these recommendations may be referred to letter ballot of the Society.

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following seven tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] These specifications and methods were accepted by the Society as tentative and appear in the 1950 Supplement to Book of ASTM Standards, Part 4.

[3] This recommendation was accepted by the Society and the revised method appears in the 1950 Supplement to Book of ASTM Standards, Part 4.

Tentative Specifications for:
Chromated Zinc Chloride (D 1032 – 49 T),[4]
Tanalith (D 1034 – 49 T),[4]
Creosote-Coal-Tar Solution (D 391 – 49 T),[4]
Zinc Chloride (D 432 – 49 T).[4]
Tentative Methods of:
Chemical Analysis of Chromated Zinc Chloride (D 1033 – 49 T),[4]
Chemical Analysis of Tanalith (D 1035 – 49 T),[4]
Chemical Analysis of Zinc Chloride (D 199 – 49 T).[4]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman).—This subcommittee contemplates further study of Tentative Methods for Establishing Structural Grades of Lumber (D 245 - 49 T) to consider possible revision and clarification in the light of data from studies under way, including factors affecting the strength of wood in compression. Study is also under way on the revision of Standard Specifications for Round Timber Piles (D 25 - 37).

Subcommittee II on Laminated Timber (F. J. Hanrahan, chairman).—Consideration will be given during the year to any comments that may be received on the new Tentative Methods of Testing the Integrity of Glue Joints in Laminated Wood Products for Exterior Service. Study will be continued on possible improvement of this method, which was based on extended tests at the Forest Products Laboratory. Data supporting this test procedure are given in a paper which was presented before the American Society of Mechanical Engineers.[6]

Subcommittee III on Plywood (J. A. Liska, chairman).—Attention will be given to the possible improvement of some of the definitions included in Tentative Definitions of Terms Relating to Veneer and Plywood (D 1038 - 49 T) and to the advancement of this tentative to standard.

Subcommittee VI on Timber Preservatives (R. H. Bescher, chairman).—The principal work of this subcommittee consisted in reviewing the recently published tentatives in conjunction with the presently made recommendations for advancement to standard, which are given earlier in this report.

Subcommittee VII on Wood Poles and Cross Arms (C. D. Hocker, chairman).—Special effort has been made during the year to publicize the proposed pole testing program, outlined by the subcommittee as a basic requirement for the rationalization of methods of tests and the development of strength data relating to specifications for treated and untreated wood poles of various species. Much consideration has also been given to methods of implementing the extended research program that has been formulated.

The work of the subcommittee has been further expanded through the establishment of a task group on crossarms. Active work is under way on the development of methods of testing crossarms by this task group, under the chairmanship of L. J. Jacobi.

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman).—The chairman reported to the committee on the International Conference on the Standardization of Methods of Testing Wood and Related Forest Products Problems, held at Geneva in 1949 under the auspices of the Food and Agriculture Organization of the United Nations.

The principal activity of the subcommittee during the year has been the development of a secondary procedure for methods of testing small clear specimens of timber involving, for the testing of material from very small trees, the possible use of specimens 1 by 1 in. in cross-section for static bending and compression parallel to grain in lieu of the 2 by 2-in. specimens. This secondary procedure was developed to give results comparable with those obtained under the primary procedure employing 2 by 2-in. specimens for all tests. It is recommended that whenever possible the procedure for the primary method be used regardless of the size of trees and that the secondary method be used only when small trees, generally less than 12 in. in diameter, are available to provide the test specimens, and only when such trees, because of crook, cross grain, knots, or other defects, are of such quality that larger clear straight-grained specimens required for the 2 by 2-in. specimens cannot reasonably be obtained.

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[4] 1949 Book of A.S.T.M. Standards, Part 4.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] T. R. Truax and M. L. Selvo, "Results of Accelerated Tests and Long-Term Exposures on Glue Joints in Laminated Beams," *Transactions*, Vol. 70, No. 4, May, 1948, pp. 393–400.

Subcommittee X on Nomenclature and Definitions (R. R. Cahal, chairman).—The work of this subcommittee has been reactivated during the year, and first consideration has been given to the revision of tree names for softwood species as presented in the Standard Definitions of Terms Relating to Timber (D 9 - 30).

Subcommittee XIII on Durability and Exposure (T. R. Truax, chairman).—The organization of this subcommittee was completed, and it is contemplated that consideration will be given to the development of methods of test for durability and exposure during the coming year.

Subcommittee XIV on Methods of Chemical Analysis (G. J. Ritter, chairman). —Subcommittee XIV was organized during the year to give consideration to methods of chemical analysis of wood, a field which has so far received little attention from Committee D-7. The subcommittee has studied the field of methods of chemical analysis and has recommended a number of procedures as tentative, as referred to earlier in this report. Work will be continued in developing further procedures during the coming year.

This report has been submitted to letter ballot of the committee, which consists of 75 voting members; 53 members returned their ballots, of whom 11 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS[1]

During the year, Committee D-8 held two meetings: one in Atlantic City, N. J., June 30, 1949, the other in Pittsburgh, Pa., on March 1, 1950.

The committee suffered the loss, through death, of three members: J. J. Shank, who died September 16, 1949; H. A. Nelson, who died March 21, 1950; and C. S. Reeve, who died in Florida, on January 6, 1950. Both Mr. Shank and Mr. Nelson were active members of Committee D-8 for many years; Mr. Shank served on Subcommittee XII, whereas Mr. Nelson served as a consulting member of Subcommittee VIII. Mr. Reeve had been a member of the Society for forty-five years and served during his time on Subcommittees I, II, V, VI, VII, VIII, IX and XI. A memorial resolution in recognition of his many years of service has been prepared by Committee D-8 and copies sent to members of his family.

J. S. Miller and H. C. Howell, who have served as chairman and secretary respectively, have found it necessary to tender their resignations as officers of the committee.

All of the standards and tentatives for which Committee D-8 is responsible have been studied and the recommendations are as set forth in this report.

NEW TENTATIVE

The committee recommends that the list of definitions developed by Subcommittee I on Nomenclature and Definitions be accepted for publication as tentative, as appended hereto:[2]

Proposed Tentative Definitions of Terms Relating to Bituminous Waterproofing and Roofing Materials.

REVISIONS OF TENTATIVES

The committee recommends that the following two tentatives be revised as indicated and continued as tentative:

Tentative Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica **(D 224 - 46 T)**[3] and *Tentative Specifications for Asphalt Roofing Surfaced with Mineral Granules* **(D 249 - 46 T)**:[3]

Section 2.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating, [compounded with a finely powdered mineral filler] *which may be compounded with a fine mineral stabilizer,* substantially insoluble in water, and finally surfaced on the weathered side with . . .

Table I.—In Specifications D 224 - 46 T, delete the 15 per cent requirement for "Percentage by weight of mineral matter passing a No. 100 (140-micron) sieve, . . ." in both the 65-lb. and 55-lb. grades. In Specifications D 249 - 46 T delete the 15 per cent requirement for

the same item in the one grade covered in the specification.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The Definitions were accepted as tentative by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

[3] **1949 Book of A.S.T.M. Standards, Part 3.**

TENTATIVE REVISIONS OF STANDARDS

The committee recommends revisions of the following three standards for publication as tentative revisions:

Standard Specifications for Asphalt Shingles With Mineral Granules (**D 225 – 46**):[3]

Section 2.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> In the process of manufacture, a single thickness of ***dry*** roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with [at least one layer of] a hot asphaltic coating, [compounded with a mineral filler] ***which may be compounded with a fine mineral stabilizer*** substantially insoluble in water, and finally completely surfaced on the weather side with granules embedded in the hot asphaltic coating.

Table I.—Delete the 15 per cent minimum requirement under "Percentage by weight of mineral matter passing a No. 100 (149-micron) sieve, . . ." for Types I, II and III.

Standard Specifications for Asphalt Siding Surfaced with Mineral Granules (**D 699 – 46**):[3]

Section 2.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on both sides with a hot asphaltic coating, [compounded with a fine mineral filler] ***which may be compounded with a fine mineral stabilizer*** substantially insoluble in water, and finally surfaced on the weather side with mineral granules embedded in the hot asphaltic coating.

Table I.—Delete the 15 per cent minimum requirement for the same item, as recommended in Specifications D 225, in the one grade covered in this specification.

Standard Specifications for Wide Selvage Asphalt Roofing Surfaced with Mineral Granules (**D 371 – 46**):[3]

Section 2.—Change the first sentence to read as follows by the addition of the italicized words and the omission of those in brackets:

> In the process of manufacture, a single thickness of dry roofing felt shall be impregnated with a hot asphaltic saturant, then coated on the weather side for approximately one-half the width of the sheet with [an asphaltic coating (compounded with a fine mineral filler substantially insoluble in water)], ***a hot asphalt coating, which may be compounded with a fine mineral stabilizer substantially insoluble in water*** and surfaced by embedding mineral granules in the hot asphaltic coating.

Table I.—Delete the 15 per cent minimum requirement for "Percentage by weight of mineral matter passing a No. 100 (149-micron) sieve, . . ." in both the 45-lb. and 55-lb. grades.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-8 recommends that the following tentatives, which have been published by the Society for two years or more, be continued without revision while being reviewed with the possibility that there will be revisions recommended before the next Annual Meeting:

Tentative Methods of Sampling Bituminous Materials (**D 140 – 49 T**),

Tentative Recommended Practice for Accelerated Weathering Test of Bituminous Materials (**D 529 – 39 T**),

Tentative Specifications for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (**D 1001 – 48 T**), and

Tentative Method of Test for Asphalt Roll Roofing, Cap Sheets, and *Shingles* (**D 228 – 48 T**).

REAPPROVAL OF STANDARDS

Committee D-8 has reviewed the following standards which have been published for six years without revision and recommends their reapproval as they conform to present practice:

Standard Specifications for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (**D 173 – 44**), and

Standard Specifications for Asphalt for Use in Constructing Built-Up Roof Coverings (**D 312 – 44**).

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be announced at the Annual Meeting.[4]

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. J. Steiner.
Vice Chairman, E. H. Berger.
Secretary, G. W. Robbins.

This report has been submitted to letter ballot of the committee, which consists of 66 members; 54 members returned their ballots, of whom 50 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. S. MILLER
Chairman.

KONRAD STERN
Acting Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS[1]

Committee D-9 on Electrical Insulating Materials held two meetings during the year and will hold a third during the Annual Meeting of the Society in June. These meetings were held in Washington, D. C., on November 28 to 30, 1949, and at Old Point Comfort, Fort Monroe, Va., on March 20 to 22, 1950.

At these meetings, action was taken on several new methods, new recommended practices, revisions in existing tentatives, and changes in some of the standards under the jurisdiction of Committee D-9. There are some projects that are still under way which will be mentioned later in the report.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. H. Scott.
Vice-Chairman, E. A. Snyder.
Secretary, M. Guarnier.

RECOMMENDATIONS ACCEPTED BY ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-9 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

New Tentative Specifications for:

Absorbent Laminating Paper for Electrical Use (D 1080 - 49 T),

New Tentative Methods of:

Test for Power Factor and Dielectric Constant of Natural Mica (D 1082 - 49 T),

Revision of Tentative Methods of:

Testing Askarels (D 901 - 46 T),

Revision and Reversion to Tentative of Standard Methods of:

Testing, Grading, and Classifying Natural Mica (D 351 - 46) in the form of Tentative, Specifications for Natural Muscovite Mica Based on Visual Quality (D 351 - 49 T).

These recommendations were accepted by the Administrative Committee on Standards on March 1, 1950, and the new and revised tentatives appear in the 1949 Book of A.S.T.M. Standards, Part 6.

Committee D-9 has approved by letter ballot during the past year several items that are under joint jurisdiction with other technical committees of the Society.

RECOMMENDATIONS AFFECTING STANDARDS

As a result of the year's work, the committee is recommending revisions of nine tentatives, tentative revisions of one standard, and revision of one standard. The standards and tentatives affected, together with recommended revisions are presented in detail in the Appendix to this report.

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (A. H. Haroldson, chairman), has continued its efforts on rewriting the Tentative Methods of Testing Varnishes for Electrical Insulating (D 115 - 48 T). The revised method will be more useful for classification, control, and definition purposes with emphasis on performance tests. Another round-robin testing program for set time of heat reactive laminating varnishes has been completed. Further study is planned to determine whether a mechanical method can be used for determining the end point to eliminate errors introduced by the technician in observing the end point of the test. The Section on Silicone Insulating Varnish has several proposed tests under consideration which will be included in future methods of tests for this type of insulating varnish.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26 to 30, 1950.

[2] **The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.**

Members of the Section on Oil Resistance met with representatives of Subcommittee IV for the purpose of cooperating on the proposed development of an improved method of evaluating the effect of varnishes on transformer oil. It was the general opinion that this matter is largely one of the performance characteristic of the varnish within the oil. For this reason, it was felt that no new test methods are necessary, since there are several methods for testing oil contamination already covered in methods of test sponsored by the Society. Apparently these have been found satisfactory. Then too, there is the question of apparatus design and construction which makes such a problem a rather complex one to evaluate in the laboratory tests. The matter has been dropped until some new plan can be devised that will permit such an evaluation.

There was a delay in the round-robin tests to evaluate a method for film continuity, draining characteristics, and moisture resistance, and also on a method for determining mechanical properties by measuring creepage and deformation on a stranded cable specimen. Plans for completing this work during the next year appear promising.

Subcommittee III on Plates, Tubes, Rods, and Molded Materials (G. H. Mains, chairman) has recommended several changes during the past year in the methods of testing under its jurisdiction. Complete texts of each of these changes appear in the Appendix to this report.

Work has been completed in connection with the development of a test method for stress relief and the effect of soldering in proximity to plastic tubing. It has been reported that work on heat loss and plasticizer migration in plastic tubing is also being actively pursued as a project under the direction of the Society of Plastic Industry. This matter will be considered when it is made available. A round-robin on flexural strength tests of thin specimens has been started to investigate several important factors involved in testing thin test specimens of sheet materials. The Standard Method of Test for Punching Quality of Phenolic Laminated Sheets (D 617 - 44) is being revised, particularly in regard to obtaining improved photographs for illustrating (*a*) surface cracks, (*b*) bulging around holes, (*c*) delamination around holes, and (*d*) drag at edges in connection with the point rating described in the method. Extensive revisions in the Standard Specifications for Laminated Thermosetting Materials (D 709 - 49 T) are being considered to bring these specifications into a more general agreement with the new Military specifications for Laminated Products and similar N.E.M.A. Standards for this product.

Interlaboratory tests on vulcanized fibre, showing how conditioning affects the dielectric strength, indicate that additional data are required to obtain equilibrium conditioning times to cover a suitable range of thicknesses of vulcanized fibre in the laboratory atmosphere (23 C., 50 per cent relative humidity).

Committee D-9 is assisting Committee D-20 on Plastics in preparing specifications for electrical grades of urea and melamine molding compounds.

Subcommittee IV on Insulating Oils (E. A. Snyder, chairman) continued its study of the evaluation of uninhibited and inhibited transformer oil.

The uninhibited mineral transformer oil studies have been a continuation of the field tests which were begun in 1944. The object of this work is to evaluate the present Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil, (D 670 - 42 T) in terms of the actual transformer use of the oil. This work comprises the study of oil in transformers which are in operation at the Detroit Edison Co., Commonwealth Edison Co., American Gas and Electric Service Corp. and at the New England Power Co. Nineteen laboratories are cooperating in this study. A summary of the results obtained were described in a paper by Frank M. Clark, "Evaluation of Mineral Transformer Oil During Service—Part I."[2] A second summary will be presented in 1951.

Four types of testing procedures have been adopted for cooperative study of inhibited transformer oil. These include a modified sludge accumulation and a high-pressure oxidation test, a modified turbine oil test, and an oxygen absorption test. The object of this study is to establish a significant reproducible test for inhibited oils which will be later evaluated in terms of oil change during actual transformer use.

Subcommittee IV is the official advisory body to the United States Committee of the International Electrotechnical Commission's Committee No. 10 on Insulating Oil. F. M. Clark, chairman of Section B, served as one of the U. S. official delegates to the international meeting of the I.E.C.'s Committee No. 10 held at Stresa, Italy, June 12 to 15, 1949. The meeting was attended by national delegates from Belgium, France, Great Britain, Hungary, Italy, Netherlands, Norway, Poland, Sweden, Switzerland, and the United States.

While many matters relating to the use of electrical insulating oils were discussed, the main objective was the development of an international oxidation stability test, or the so-called "sludge test" for transformer oils with the ultimate goal of establishing international specification values. A committee was organized to direct a series of cooperative international test studies directed toward the final establishment of an international method to determine the oxidation stability of electrical insulating oils. Mr. Clark was appointed as U. S. representative. Representatives from England, Holland, Switzerland, France, and Italy were also appointed.

During the year, numerous points that require study have arisen in connection with the Standard Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin (D 924 - 49). The entire method was reviewed and a revision is being prepared. A new test method to determine the insulation resistance of insulating oils is also being prepared.

A study to determine the necessary modifications required in the Method of Test for Dielectric Strength of Insulating Oil of Petroleum Origin (D 877 - 49) so that it can be used on high viscosity oil is under way.

After a rather extensive study of methods to determine the percentage of water present in insulating oils, considerable difficulty was experienced in securing satisfactory and accurate check tests. Consequently, the subcommittee is not ready to offer a tentative test method.

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[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 1041 (1949).

Revisions are recommended in the Methods of Testing Askarels, (D 901 - 49 T) as given in the Appendix. Methods for determining possible contamination of askarels by oils or various types of solid insulations are being studied.

Tests for Electrical Insulating Oils (D 117 - 47 T) have been completely revised and several test methods, not previously included, have been added. A complete text of the revision is appended hereto.[3]

The Method of Test for Interfacial Tension of Oil Against Water by the Ring Method (D 971 - 48 T) has been approved as a desirable test for use on insulating oils and is now included in the Tests for Electrical Insulating Oils (D 117 - 50 T).[3]

Preparation of a method of test to detect the presence of corrosive sulfur (from free and combined sulfur) in electrical insulating oils is practically completed. There remains, however, development work on a procedure to give a quantitative measure of the amount of such corrosive sulfur present.

No symposia on insulating oils were held during the year, but reprints of the papers and complete discussions of the March, 1949, symposium were made available as Special Technical Publication No. 95. The papers included in the publication are: F. M. Clark, "Performance Characteristics of Askarels," R. G. Call, "One System of Laboratory Testing to Appraise the Serviceability of Oils in Transformers," and G. H. von Fuchs, "Performance of Inhibited Transformer Oils."

Subcommittee V on Ceramic Products (K. G. Coutlee, chairman) has made several significant recommendations relative to the testing of steatite and glass-bonded mica electrical insulation during the year. These are based on rather extensive round-robin power factor and dielectric constant tests at 1 megacycle and direct-current insulation resistance tests on pairs of disk test specimens in accordance with JAN-I-10 connected in parallel in sandwich form, after prolonged exposure to and while under the condition of 90 per cent relative humidity at room temperature. The following conclusions were made:

1. Lack of satisfactory interlaboratory agreement obtained on the power factor and dielectric constant test results on both steatite and glass-bonded mica under the conditions stated above prompted the subcommittee to recommend that these two materials should be tested for power factor and dielectric constant in the dry state, in accordance with Procedure A of Conditioning Methods D 618 for purposes of establishing loss factor classification.

2. Direct-current insulation resistance, volume resistivity, and surface resistivity shall be determined in accordance with Methods D 257 after an exposure of 96 hr. and while still under the condition of 90 per cent relative humidity at 35 C. (Procedure C of Methods D 618) at 100 v. d.c. and an electrification time of 1 min.

3. In special cases, it may be of interest to study the behavior of power factor, dielectric constant, and d-c. insulation resistance of steatite and glass-bonded mica under high humidity, using air-drying silver paint electrodes which are sufficiently porous to permit diffusion of moisture through them into the body of the material as a means of determining electrical stability under such conditions.

Data substantiating these recommendations can be found in subcommittee minutes.

There is a need for revision of several

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[3] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Parts 5 and 6.

of the test methods for ceramic products that have been published for years. This task has become increasingly difficult, due to the apparent lack of interest in the work of the subcommittee. It was suggested that joint meetings of the subcommittee with Committees C-21 on Whitewares and C-14 on Glass and Glass Products at least once a year might stimulate interest in the work of Subcommittee V.

Subcommittee VI on Solid Filling and Treating Compounds (W. A. Evans, chairman) has completed round-robin tests on microcrystalline waxes. The test methods are being prepared for presentation to Committee D-9 for letter ballot. Plans are under way to prepare specifications for microcrystalline waxes upon the completion of the test methods. The subcommittee is also planning a revision of the Tests for Solid Filling and Treating Compounds (D 176 - 44), excluding the methods applicable to microcrystalline waxes.

There appears to be an insufficient interest in a test for oil solubility determination. For this reason it was decided to make the Section on Test Methods for Oil Solubility Determination inactive for the present.

Subcommittee VII on Varnished Fabrics (R. W. Chadbourn, chairman) recommended changes in the Specifications for Black Bias-Cut Varnished Cloth Tape (D 373 - 49 T) during the year. These changes involved the listing of tapes as 12-mil, 10-mil, and 7-mil to replace the present indefinite listing. The present listing provides for tapes of less than 7 mils thickness, which is beyond the written scope of the specifications. Also, tapes are usually made in and designated by the above thickness values. The subcommittee is still collecting data on the dielectric strength of varnished cloth tapes under 6 per cent elongation, relative to revising the specification requirements to meet the present condition of manufacture. Round-robin tests have been instituted to determine what change may be necessary in the present temperature elongation formula in Specifications D 373. A new method for insulation resistance is under consideration, along with numerous other changes being made in Tentative Methods D 295 and Specifications D 373.

Significance statements have now been adopted for most of the tests in Tentative Methods D 295 (Varnished Cloths and Tapes) and Tentative Methods D 902 (Varnished Glass Cloths and Tapes). With the addition of one or two more statements now under consideration, this work will be completed.

Round-robin tests on resistance to heat aging are being conducted on both oleoresinous and silicone-varnished glass fabric samples.

An agreement has been reached between Committees D-9 and D-11 on Rubber and Rubber-Like Materials concerning development of test methods for plastic pressure-sensitive tapes. Committee D-9 will develop test methods, with Subcommittee IX of Committee D-11 having representation on the D-9 section while this work is in progress.

Subcommittee VIII on Insulating Papers (E. G. Ham, chairman) has recommended several additions to D 202 for insulating papers. The text of these new tests is appended to this report.[4] A method for measuring sulfate residues in paper which is a TAPPI Standard (T 468) was found suitable for such determinations of insulating papers. The test method was rewritten in the Society's recommended form and accepted by the committee for adoption. Methods for determining the extent of water and alcohol soluble matters in insulating paper were also adopted for inclusion in Methods D 202. These test methods had previously been in use by several members of the subcommittee for several years and had been found satisfactory. A method for the determination of fiber composition of paper has been absent from the Society's standards for several years. Recently, Committee D-6 on Paper and Paper Products adopted a Method of Test for Fiber Analysis of Paper and Paperboard (D 1030 - 49 T). The method was found suitable for insulating papers. For this reason, reference to its use is now to be made a part of Methods D 202.

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[4] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Parts 4 and 6.

After completing the Specifications for Absorbent Laminating Paper (D 1080 - 49 T) the subcommittee was requested to draw up specifications for interlayer type of insulating papers.

Some of the tests in Methods D 202 have now been amplified by the addition of significance statements. Additional statements of significance are being written for the remainder of the tests within D 202.

Changes in the sections of D 202 covering the deterioration test have been revised so that more sensitive methods for measurement of heat stability will be specified. A reduction in the severity of the heat treatment which may show relative permanence was also included in the revision.

Subcommittee IX on Mica (E. O. Hausmann, chairman) recommended the following revisions of Standard D 351 - 46:

1. Changing the visual quality classification table to a form that is mutually agreeable to all mica interests,
2. Adding color transparencies as standards of quality.
3. Removing the power factor and dielectric constant test and issuing it as a separate tentative method, and
4. Adding an appendix describing a method for optical axial angle of mica.

The first two items are matters of agreement on definitions in quality and, thus, no further justification is required. The removal of the power factor and dielectric constant test from the standard was largely an editorial change. The subcommittee recommended the addition of the optical axial angle between the limits of 50 and 75 deg. as a test for identifying Muscovite mica.

Subsequent to action by Committee D-9, it was found that color transparencies would not be available, so action was taken to remove all reference to color transparencies from the method. This was considered to be an editorial change. Subcommittee IX has been very actively trying to get suitable color transparencies. Earlier hopes for obtaining satisfactory transparencies have diminished very decidedly and there is serious doubt now that they can be produced, although efforts are still being made.

The subcommittee provided for representation of American mica interests at the meeting of a Committee on Mica of the International Standards Organization (ISO/TC56) in Delhi, India, last January. Although action of that committee was largely in agreement with American ideas, there was disagreement with certain actions. These items are still under discussion.

Subcommittee XI on Significance of Tests (J. H. Palmer, chairman) submitted to the committee a Proposed Recommended Practice for Writing Statements as to the Usefulness of Tests of Electrical Insulating Materials. This was approved by a letter ballot of the committee and appears in the January, 1950 compilation of D-9 standards. It is now unnecessary for the subcommittee to approve significance statements if the proposed recommended practice is followed.

Subcommittee XII on Electrical Tests (K. N. Mathes, chairman) made several editorial changes in the Methods of Test for Electrical Resistance of Insulating Materials (D 257 - 49 T). A bibliography for D 257, with references appropriately located throughout the text, is being prepared.

Work on arc resistance is continuing. Two section meetings were held during the past year. At both meetings, the results of the round-robin completed by the eight participating laboratories on Methods D 495 were discussed. Statistical analysis of the data indicated the over-all average relative variation for eight laboratories and for all six materials to be about 20 per cent. While this result was somewhat disappointing, the general opinion is that the present method is still a definite improvement over the previous method. A more limited round-robin is planned to investigate the reasons for disagreement on the results obtained for some special "arc-resistant" materials.

Task groups are at work studying: the extension of Methods D 495 to include higher current arcs (above 40 ma.), the development of a method of providing a continuous rate of increase in arc severity, and other forms of arc-resistance tests.

The subject of suitable electrode configurations and electrode materials is being actively pursued toward preparing statements relative to application techniques and their uses, where appropriate, in the various electrical test methods.

A revision of Method D 150 for Power Factor and Dielectric Constant is under way. The revised method will include methods for measurements of a-c. loss characteristics at ultra-high frequencies.

Subcommittee XIV on Conditioning (A. C. Webber, chairman) is a joint subcommittee of Committees D-9 and D-20. Revisions of Conditioning Methods D 618 are being prepared by the subcommittee to include additional standard elevated test temperatures together with workable tolerances, along with several other changes that are editorial in nature. Editorial changes in the Recommended Practice for Maintaining Constant Relative Humidity by Means of Aqueous Solution (D 1041 - 49 T) were recommended. These changes will delete unnecessary terms and insignificant figures in the formula used for calculation of concentration of solates in the humidity controlling solutions.

One of the more important items under consideration of the subcommittee is relative to the revision of Specifications D 760 and D 761 for Servicing Units and Enclosures for Tests of Electrical Insulating Materials and Plastics at Other than Normal Temperatures. The reasons expressed for the need of these revisions were that the present specifications are: (*a*) too inflexible, such as constructional details that should be left to the discretion of the designer are rigidly prescribed, and (*b*) do not include adequate performance requirements.

Subcommittee XIII on Mechanical Tests is temporarily inactive. Such mechanical methods of test, involving tests under the committee's jurisdiction, receive consideration within the various subcommittees.

This report has been submitted to letter ballot of the committee, which consists of 167 voting members; 79 members returned their ballots, of whom 75 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. T. HATCHER,
Chairman.

E. A. SNYDER,
Vice-Chairman.

O. E. ANDERSON,
Recording Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this appendix are given recommendations affecting certain standards and tentatives concerning electrical insulating materials which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1949 Book of A.S.T.M. Standards, Part 6, and also in the January, 1950 Compilation of A.S.T.M. Standards on Electrical Insulating Materials.

Revision of Tentatives

Tentative Specifications for Orange Shellac and Other Indian Lacs for Electrical Insulation (D 784 - 44 T):

These revisions were recommended because it was the concensus of the members that the present specifications provide ranges of test temperatures that are beyond the general practice followed in other A.S.T.M. specifications:

Table I.—Change the heading "Polymerization time" to read "Polymerization time at 150 ± 1 C." and the heading "Flow test" to read "Flow test at 110 ± 1 C."

Tentative Specifications for Black Bias-cut Varnished Cloth Tape Used for Electrical Insulation (D 373 - 49 T):

The revision of these specifications replaces the indefinite listing of tape thicknesses with three specified nominal thicknesses, within the scope of the specifications. The thicknesses selected have become universally accepted as standard for this type of electrical insulation:

Table I.—Under the heading "Nominal Thickness", list "12 mils," "10 mils," and "7 mils," respectively in place of the present values.

Section 10 (a).—In the third sentence change the phrase "from 5 to 9 mils in thickness" to "of 7 mils nominal thickness"; change the phrase "10 to 12 mils in thickness" to "of 10 and 12 mils nominal thickness."

Tentative Methods of Testing Nonrigid Polyvinyl Tubing (D 876 - 46 T):

This revision provides for a measure of internal stress retained in plastic tubing after extrusion and indicates to some extent the amount of shrinkage which may occur when the tubing is in close proximity to a joint being soldered or when an assembly is exposed to heat in the process of manufacture.

New Sections 50 to 53.—Add the following new test procedure for internal stress measurement:

Internal Stress Measurement

50. *Apparatus.*—The apparatus shall consist of the following:

(*a*) A covered stainless steel or heat resistant glass tank at least 12 in. long by 5 in. wide and 5 in. deep.

(*b*) A basket of lightweight stainless steel wire screening at least 11 in. long approximately 1 in. deep and of a width slightly less than the width of the tank. It shall be compartmented with screening in a lengthwise direction to hold tubing specimens straight while immersed. A wire-screen cover shall be provided to keep specimens in their respective compartments and to insure complete immersion.

(*c*) A source of controlled heat.

(*d*) A thermometer calibrated to not less than 1 C. per division.

(*e*) A scale graduated to 0.01 in.

51. *Test Specimens.*—Three straight lengths of tubing 10 ± 0.01 in. long and cut square at the ends.

52. *Procedure.*—Fill the tank with glycerine to a level about 1 in. below the top and with the basket immersed, bring the temperature of the glycerine up to a steady state of 150 C. Raise the basket to the surface of the glycerine and place the tubing specimens in the basket as rapidly as possible not more than one specimen to a compartment. Cover the basket and lower 1 in. below the surface of the liquid for 15 min. Maintain the glycerine at 150 ± 1 C. during the entire test period. Remove the basket from the liquid and allow the specimens to cool to room temperature. Then remove the specimens from the Basket and measure the length of each.

53. *Report.*—The report shall include the following:

(*1*) Size of tubing from which specimen was taken,

(*2*) Length of tubing after completion of test,

(*3*) Percentage change in length (shrinkage) calculated on 10-in. length, and

(*4*) Average shrinkage.

Tentative Methods of Testing Electrical Insulating Oils (D 117 - 47 T):

The revision of these methods consists of several changes in the existing tests covered in this tentative as well as providing a test for interfacial tension of oil against water by the ring method. The revised methods of test, appended hereto,[1] are recommended for publication as tentative.

Tentative Methods of Testing Askarels (D 901 - 49 T):

These revisions provide for a reference to modifications of certain test procedures in the scope, improvement of the sampling procedures, and the addition of two new sections describing the determination of dielectric strength:

Section 1.—Add a second sentence to read as follows: "For askarels having a Saybolt universal viscosity of over 110 sec. at 100 F., modifications of certain test procedures are necessary."

Sections 7 (*c*) *and 8* (*c*).—Delete those paragraphs which require the rinsing of the sample container and sampling apparatus with the liquid to be sampled because Section 5, detailing the cleaning procedure, makes no further rinsing necessary. Reletter subsequent paragraphs accordingly.

Section 7 (*e*).—Delete "preferably on their sides" in the first sentence because present-day drums may have their bungs on either the side or on the top.

Section 29 (*b*).—Revise to read: "(*b*) *Water Supply.*—A water supply, the temperature of which may be varied."

New Sections 32 and 33.—Add two new sections to the method under the heading "Dielectric Strength." Delete the present note under "Dielectric Strength."

32. Except as stated in Section 33, the dielectric strength of askarels shall be determined in accordance with the Standard Method of Test for Dielectric Strength of Insulating Oils of Petroleum Origin (A.S.T.M. Designation: D 877).

33. For referee tests, breakdowns shall be made in an atmosphere having a relative humidity not exceeding 55 per cent. At higher humidities, the dielectric strength of the test samples may be materially lowered by the absorption of atmosphere moisture.

Tentative Methods of Testing Glass-Bonded Mica for Electrical Insulation (D 1039 - 49 T):

These revisions provide for additional information relative to each of the individual tests concerning test specimen sizes, conditioning procedures and other pertinent information such as electrodes and rate of voltage rise in the dielectric strength test. The revised methods of test, appended hereto,[2] are recommended for publication as tentative.

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[1] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Parts 5 and 6.

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 – 49 T):

This revision simply provides a procedure for selecting samples of varnished cambric tape shipped packed in oil.

Section 3 (*a*).—Add the following sentence after the first sentence: "On tape shipped packed in oil, the outer layer shall be discarded before samples are taken."

Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (D 902 – 47 T):

These revisions provide significance of test statements for the thickness, breaking strength, dielectric strength, resistance to oil, weight, and threads per inch tests.

New Sections.—Add the following as new Sections 6, 7, 10, 11, 16, 17, 33, 34, 41, 42, 44 and 45, renumbering the subsequent sections accordingly:

6. *Definition.*—Thickness of varnished glass cloth and varnished glass cloth tape is the perpendicular distance between the outer varnished surfaces of the material as determined in accordance with this method.

7. (*a*) *Significance.*—This test is of value in determining whether the material meets specified tolerances for thickness. In addition, thickness values are essential because of the importance of space factor in designing electrical equipment.

(*b*) Determination of dielectric strength, usually expressed in volts per mil, also necessitates thickness measurements.

10. *Definition.*—The breaking strength of varnished glass cloths and varnished glass cloth tapes may be defined as the force required to break the cloth or tapes when tested under certain prescribed conditions.

11. *Significance.*—The breaking strength of finished cloth and tape is of importance as a measure of its ability to withstand reasonable pulling without failure while being applied in service.

16. *Definition.*—The dielectric strength of varnished glass cloth or tape may be defined as the maximum potential gradient, usually expressed in volts per mil, that the material can withstand without rupture, when the cloth or tape is electrically stressed between two electrodes under prescribed conditions.

17. *Significance.*—Dielectric strength of varnished glass cloth or tape insulating material is of significance for the following reasons:

(*a*) Insulating materials are subjected to electrical stresses in service for long periods of time. Although these service stresses are usually a small fraction of the breakdown stresses determined by dielectric strength tests, it has been found that, for any given material, the service stresses which it can withstand during its life bear some relation to the breakdown stresses obtained in the dielectric strength test. This test, therefore, gives some indication of the ability of the cloths or tapes to withstand the service stresses to which they are subjected.

(*b*) This test often detects the presence of defects in the cloth or varnish such as blisters, pin-holes, foreign particles, surface checks, etc., although it is inadequate for this purpose since only a small part of the surface is explored.

(*c*) This test is of value in determining the ability of the cloth or tape to withstand conditions of elongation and hot oil, encountered in some service applications, without excessive loss of dielectric strength.

(*d*) Three methods of testing for dielectric strength are given, the "short-time," the "step-by step" or "slow-rate-of-rise," and the "endurance" tests. Choice of the method should be based on whether or not the effect of time under stress is considered an important factor, and the available time which can be allowed for each test. See also the general statement on Significance of the dielectric strength test.

33. *Definition.*—The oil resistance of varnished glass cloth or tape is the property of the varnish film to withstand the attack of mineral oil without excessive impairment of its physical and electrical characteristics when the varnished cloth or tape is immersed in a specified oil for a prescribed period of time at a given temperature.

34. *Significance:* (*a*) The oil resistance of varnished glass cloth or tape determines the suitability of the insulation for use in oil-immersed apparatus, such as oil-filled transformers and switches, and in electric cables and cable

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[2] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 6.

splices. It will also serve to indicate serviceability in proximity to lubricating oils. Such oils, however, usually have very little effect on a varnish film as compared with transformer oil.

(*b*) When immersed in oil, black varnish films usually soften and swell slightly but they should not blister, wrinkle, nor separate from the cloth. Yellow varnish films are much more oil resistant than black films and soften and swell very little, if any. In either case, the dielectric strength should not be impaired.

41. *Definition.*—The "weight" of varnished glass cloth and varnished glass cloth tapes is the weight per unit area as determined in accordance with this method. It is usually expressed in pounds-per-square-yard for a specified nominal thickness.

42. *Significance.*—The ratio of varnish weight to glass cloth weight, within and between shipments, can be determined from the weight of varnished glass cloth and varnished glass cloth tape and the weight of the cloth base. This ratio is a factor in determining the electrical characteristics of the material. Weight values are useful for estimating weight in designing electrical equipment containing a constituent part of varnished cloth or tape.

44. *Definition.*—The threads per inch of varnished glass cloth refers to the count of the number of warp and filling yarns present in the base glass cloth per linear inch of length or width.

45. *Significance:* (*a*) Thread count, together with the weight and the width of the glass cloth, is accepted as the common means for designating and identifying cloth constructions.

(*b*) Certain of the physical and electrical properties of woven fabrics are dependent on thread count. That is, assuming the same size of yarn, an increase in thread count increases the weight, breaking strength and density of the cloth. Also, the dielectric strength and power factor of the varnished fabric may be changed by altering the number of threads per inch of the cloth.

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (D 202 - 49 T):

These revisions provide for: the addition of significance of test statements for thickness, bursting strength, folding endurance, air resistance and impregnation time; the addition of the newly developed tests for water soluble matter, alcohol soluble matter, and water soluble sulfates; the reference to a fiber analysis method; and revisions in the deterioration test (heat stability test). The proposed revisions are appended to this report.[3]

TENTATIVE REVISIONS OF STANDARDS

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 49):

The following revision of the existing tentative revision of this standard provides for testing materials $\frac{1}{16}$ in. and over in thickness in accordance with A.S.T.M. Method D 790.

Sections 7 to 10.—Change the heading for those sections on flexural strength test to read, "For Materials under $\frac{1}{16}$ in, in Thickness" retaining the present footnote 5.

New Section.—Add the following as a new Section 11, renumbering the subsequent sections accordingly:

11. *For Materials $\frac{1}{16}$ in. or Over in Thickness:* (*a*) Except for definitions of cutting directions of specimens as specified in Section 2 and for conditioning as specified in Section 3, the flexural strength properties including modulus of rupture and modulus of elasticity in bending shall be determined in accordance with the Tentative Method of Test for Flexural Properties of Plastics (A.S.T.M. Designation: D 790).

(*b*) Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested for flatwise test. Where edgewise tests are desired, five specimens shall also be cut lengthwise and five crosswise of the sheet.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Standard Methods of Testing Shellac Used for Electrical Insulation (D 411 - 44):

These revisions are consistent with the reasons stated for the recommended changes given above in the Specifications for Shellac (D 784 - 44 T).

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[3] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Parts 4 and 6.

Section 3 (b).—Change the first sentence to read: "An oil bath of such construction as to permit the maintaining of a test temperature of 150 ± 1 C."

Section 6 (a).—Change the second sentence to read: "The tube shall then be inserted and held securely in a vertical position in the test rack, placed in the oil bath having a test temperature of 150 ± 1 C. maintained throughout the test." Also delete the note appearing in this section.

Sections 9 (a) and (b).—Change to read as follows:

9. (*a*) The purpose of this test is to determine the flow of shellac when subjected to 110 ± 1 C. under the conditions of test described herein.

(*b*) The method consists in melting a sample of ground shellac in a graduated test tube and then tilting the tube to a definite angle while maintained at 110 ± 1 C. in order to permit the shellac to flow down the tube.

Section 12 (b).—Change to read: (*b*) The testing fixture, with the glass test tubes in a vertical position, shall be inserted in the oil bath maintained at the test temperature of 110 ± 1 C." Also delete the note appearing in this section.

REPORT OF COMMITTEE D-10
ON
SHIPPING CONTAINERS[1]

Committee D-10 held two meetings during the year: a fall meeting in Detroit, Mich., on October 6 and 7, 1949, and a spring meeting in Madison, Wis., on April 27 and 28, 1949. There was an attendance of 34 and 53, respectively, at these meetings. Following custom, an address and an inspection trip were scheduled for each meeting. These included a talk by L. J. Markwardt, Vice-President of the Society, and personally conducted tours through the Cadillac Motor Plant and the U. S. Forest Products Laboratory.

Due to the expanding interests of the committee, a revised scope has been discussed with the Board of Directors of the Society. This broader coverage will more fully cover the entire field of packaging, including packages, packing shipping containers and pallets.

At the spring meeting, officers for the ensuing term of two years were elected as follows:

Chairman, T. A. Carlson.
Vice-Chairman, R. C. McKee.
Secretary, E. R. Stivers.
Advisory Committee: H. A. Bergstrom, A. V. Grundy.

The committee now consists of 80 members, of whom 32 are classified as consumers, 28 as producers and 20 as general interest members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the proposed Tentative Method of Test for Water Vapor Permeability of Shipping Containers (D 1008 - 49 T). This recommendation was accepted by the Standards Committee on September 22, 1949, and the new tentative appears in the 1949 Book of A.S.T.M. Standards, Part 4.

On March 1, 1950, the Administrative Committee on Standards accepted the recommendation of Committee D-10 that the Method of Testing Large Shipping Cases and Crates be published as tentative. The new tentative will appear in the 1950 Supplement to Book of A.S.T.M. Standards, Part 4, bearing the designation D 1083 - 50 T.

ADOPTION OF TENTATIVES AS STANDARD

Tentative Method of Drop Test for Bags (**D 959 - 48 T**).[2]—Committee D-10 recommends that this method be approved for reference to letter ballot of the Society, with the following revisions:

Section 5(a).—Revise to read as follows:

> 5. (*a*) *Procedure A.*—Bags shall be dropped on any face, butt or side in accordance with Paragraph (*c*).
>
> (*b*) *Procedure B.*—Bags shall be dropped on faces, butts, and sides in a specified sequence of drops as follows: (1) front (2) back (3) right side (4) left side and optional (5) bottom (6) top and in accordance with Paragraph (*c*). Before each drop the contents shall be uniformly distributed throughout the bag.
>
> (*c*) The bag shall be positioned with the

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 4.

center of gravity of the contents over the point of impact, and the face on which the bag is to fall shall be parallel to the floor. Drops shall be vertical, free and unobstructed.

Section 5(b).—Reletter as Paragraph (*d*), relettering subsequent paragraphs accordingly and revise as follows:

> (*d*) *Height of Drop.*—Drop on faces, butts and sides may be from the same height, or drops on butts and sides may be from lesser heights, in a definite relationship. Unless otherwise specified, the height of face drops shall be 4 ft.

Section 6.—Delete (*4*) and replace with the following: "(*4*) Type of apparatus and procedure used."

Tentative Definitions of Terms Relating to Shipping Containers (**D 996 - 48 T**).[2]—The committee recommends that these definitions be approved for reference to letter ballot of the Society for adoption as standard.

Tentative Method of Drop Test for Cylindrical Shipping Containers (**D 997 - 48 T**).[2]—The committee recommends that this method be approved for reference to letter ballot of the Society for adoption as standard.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions in the following standard and accordingly asks for a nine-tenths affirmative vote in order that these recommendations may be referred to letter ballot of the Society:

Standard Method of Incline Impact Test for Shipping Containers (**D 880 - 47**).[2]—For the purpose of clarification on construction of apparatus the following revisions are made:

Section 2(a).—Change second sentence to read as follows by the addition of the italicized words:

> The bumper shall be a wood barrier *with a face made of Group IV Woods of sufficient size to permit full contact with shipping container, constructed at the bottom of incline,* with the plane of the face perpendicular to the direction of the movement of carriage.

Change the fourth sentence to read as follows by the addition of the italicized words:

> The track shall accommodate the flat-bed rolling carriage or dolly which is equipped with steel wheels and a renewable face *made of Group 4 Woods* or *Group 4 Plywood.*

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definition of Terms (E. Dahill, chairman) has been working on additional terms on which there are differences of opinion. The present Tentative Definitions D 996 - 48 T was the result of nearly five years' work, indicating how such matters move. These are now being advanced to standard.

Subcommittee II on Methods of Test (E. R. Stivers, chairman) has been active on new as well as old standards. Its Section 6 did exceedingly well in obtaining approval of the new Tentative Method of Testing Large Shipping Cases and Crates in two years. The new Section 7 on Pallets in six months brought in a proposed tentative which was the center of discussion at the Madison meeting. It is expected that with some changes this proposal will be accepted within the coming year.

Subcommittee IV on Performance Standards (J. H. Toulouse, chairman) is the center of interest. This committee has several sections considering standards for various tests. These sections are finding it necessary to make a careful study of the test methods in order to more clearly evaluate the results. Since there are wide differences of opinion in regard to performance requirements, this committee has a task which is difficult and tedious. The fact that there are so few accepted standards makes its work of prime importance.

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[3] The letter ballot on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

One section has recommended weight classifications which met with general approval as follows:

	0 to 50 lb.	One man throwing
Over	50 to 90 lb.	One man lifting
Over	90 to 200 lb.	Two men lifting
Over	200 to 500 lb.	Manual handling with equipment
Over	500 to 1000 lb.	Power handling
	Over 1000 lb.	Special handling

Subcommittee V on Correlation of Tests and Test Results (R. C. McKee, chairman) is considering a series of round robin tests to determine variations in drum tests and test results, using A.S.T.M. Standard Method D 782 - 47. It is hoped this project will be under way in the near future.

Subcommittee VI on Interior Packing (W. B. Lincoln, Jr., chairman) has compiled a list of 180 terms which is ready for transmission to Subcommittee I. This has been a long, difficult task and the committee is now ready to move aggressively on other items on its docket.

This report has been submitted to letter ballot of the committee, which consists of 80 members; 58 returned their ballots, of whom 56 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

T. A. CARLSON,
Chairman.

EARL R. STIVERS,
Secretary.

REPORT OF COMMITTEE D-11

ON

RUBBER AND RUBBER-LIKE MATERIALS[1]

Committee D-11 on Rubber and Rubber-Like Materials held one meeting during the year in Pittsburgh, Pa., on March 3, 1950, in connection with the spring group meetings of A.S.T.M. committees. All of the subcommittees, however, have been actively carrying on their work throughout the year by correspondence and in many cases, have held separate meetings.

Mention was made in the 1949 report of an invitation which had been extended to ISO Technical Committee 45 to meet in the United States in 1950. Since that time, plans have developed for an international rubber conference to be held in Cleveland during the week of October 9 under the sponsorship of the Rubber Division of the American Chemical Society. Since this will bring a large number of foreign guests to this country, ISO Technical Committee 45 has accepted the invitation and has designated the week following the international conference as the time for its meetings. Committee D-11 will act as host for the meetings of this ISO Technical Committee for which plans are now in preparation.

The special committee within Committee D-11 on standard reference samples of rubber compositions and rubber compounding ingredients, which has been cooperating with the National Bureau of Standards in the development of this standardization program, has continued its work of securing agreement on a series of typical rubber compound formulations from which standardized rubber samples may be made available. Proposed formulations have been circulated for criticism and are practically ready for formal recommendation. One factor which has retarded this program has been the difficulty in obtaining an adequate supply of a uniform standard crude rubber for use in the formulations.

The election of officers for the ensuing term of two years resulted in the reelection of the following:

Chairman, Simon Collier.
Vice-Chairman, H. G. Bimmerman.
Secretary, Arthur W. Carpenter.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-11 submitted the following recommendations to the Society through the Administrative Committee on Standards:

New Tentative Specifications and Methods of Test for:

Concentrated, Ammonia Preserved, Creamed and Centrifuged Natural Rubber Latex (D 1076 - 49 T).

New Tentative Method of:

Test for Evaluating Pressure Sealing Properties of Rubber and Rubber-Like Materials (D 1081 - 49 T), and

Test for the Curing Characteristics of Vulcanizable Mixtures during Heating by Means of Shearing Disk Viscometer (D 1077 - 49 T).

Revision of Tentative Method of:

Test for Compression Set of Vulcanized Rubber (D 395 - 47 T),

Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (D 471 - 46 T),

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

Testing Asphalt Composition Battery Containers (D 639 – 46 T),

Test for Indentation of Rubber by Means of a Durometer (D 676 – 47 T), and

Test for Viscosity of Rubber and Rubber-Like Materials by the Shearing Disk Viscometer (D 927 – 47 T).

Revision of Tentative Specifications and Methods of Test for:

Latex Foam Rubbers (D 1055 – 49 T), and

Sponge and Expanded Cellular Rubber Products (D 1056 – 49 T).

Tentative Revision of Standard Methods of:

Testing Compressed Asbestos Sheet Packing (D 733 – 49).

Revision and Reversion to Tentative of Standard Methods of:

Tension Testing of Vulcanized Rubber (D 412 – 41 T).

These recommendations were accepted by the Standards Committee on December 22, 1949, with the exception of the last recommendation, which was accepted on January 17, 1950. The new and revised tentatives and the tentative revision of Standard D 733 appear in the 1949 Book of A.S.T.M. Standards, Part 6.

The new Tentative Specifications and Methods of Test for Natural Rubber Latex were originally published as proposed specifications with the 1949 Annual Report, but the committee decided to give them tentative status at once, after agreement was reached on controversial points, since they are urgently needed for adequate control of the quality of natural rubber latex coming into the market.

The new Tentative Method of Test for Evaluating Pressure Sealing Properties of Rubber has been used by the U. S. Navy for some time for the evaluation of packings, and the committee has prepared the method in standardized form in the belief that it is valuable for use in many other applications of rubber. The new Tentative Method of Test for Determining the Curing Characteristics of Vulcanizable Mixtures by Means of the Mooney Viscometer has been found to be valuable for studying the scorching characteristics of compounds during processing and for evaluating the rate of cure.

The committee has been working for several years to standardize Methods for Testing Rubber Thread since no generally accepted standard procedures have been available in this field. It was considered advisable to make available the results of this work through publication of those tests which have been agreed upon in the form of a proposed method of test. Work will continue on the evaluation of the proposed methods and on the possible addition of others, but it is expected that the present publication will greatly aid the committee by stimulating wider use and suggestions.

The revision of Methods D 471 improved the definition of the composition of the standard reference fuels used as immersion liquids and the method for removing immersion oils from specimens after immersion. The revision of Method D 395 for Compression Set Determination contained editorial corrections and changed the conditions for heat treatment of the specimens to include a higher temperature than previously was permitted but which is in common use in the industry. In the Hardness Test Method using a Durometer, a number of details of technique have been in controversy for an extended period. Agreement has been reached in the committee concerning these matters and the revision incorporates these agreements in the tentative methods. In the 1949 Annual Report, the Specifications and Methods of Test for the different types of Cellular Rubbers were completely revised and separated. Following publication of these changes, need for additional revision became evident and was effected by the recommendations accepted by the Standards Committee. The revision of the Test Methods for Asphalt Composition Battery Containers applied only to the preparation of the test specimen for the acid absorption test. In the case of the Mooney Viscosity Method, D 927, a number of test details were clarified and better defined. This applied particularly to control of temperature conditions. The change in the Standard Method of Testing Compressed Asbestos Sheet Packing, D 733, involved only the addition of an explanatory note. In the case of the editorial note added in the Oxygen Bomb Aging Method and the Air Pressure Heat Test, an important cautionary note was added concerning the danger of using oil or other combustible organic fluids as heating media where oxygen or high pressure air might be involved. The reversion to tentative and the revision of the Standard Methods of Tension Testing, D 412, brought into published form the results of several years' work by the committee, incorporating into these methods new developments and improvements which have resulted from experience in connection with the use of synthetic rubbers.

The four new Tentative Specifications on Rubber Protective Equipment, accepted last June, were approved by the American Standards Association on January 9, 1950, and carry the following ASA designations:

Specifications for Rubber Insulating Line Hose (D 1050 - 49 T; ASA J6.1-1950)
Specifications for Rubber Insulator Hoods (D 1049 - 49 T; ASA J6.2—1950)
Specifications for Rubber Insulating Blankets (Without Fabric Reinforcement) (D 1048 - 49 T; ASA J6.4—1950), and
Specifications for Rubber Insulating Sleeves (D 1051 - 49 T; ASA J6.5-1950.)

On recommendation of Committee D-11, Gordon Thompson has been appointed A.S.T.M. representative in ASA Sectional Committee C-8 on Insulated Wires and Cables, replacing F. M. Farmer. W. P. Tyler has been appointed as Committee D-11 representative in the new Committee E-13 on Absorption Spectroscopy. G. H. Swart has been designated to represent Committee D-11 in Committee D-19 on Industrial Water. Committee D-11 is interested in the work of Committee D-19 on the development of specifications for high purity water for use as a chemical reagent and in certain process work since this is important in the manufacture of synthetic rubbers.

REVISIONS OF TENTATIVES

Although the committee is not proposing any new tentatives at this time, it is recommending that nine of the present tentatives be revised as indicated and continued as tentative:

Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (**D 27 - 46 T**).[2] —This revision includes minor changes to bring this specification into conformity with present practice:

Section 6.—Change the value of "1 per cent under" to "1 per cent under for solid conductors and 2 per cent under for stranded conductors" in the permissible variations specified in this section. This is necessary to conform with Standard Specifications for Concentric-Lay-Stranded Copper conductors, Hard, Medium-Hard or Soft (B 8 - 49).

Section 7 (a).—Change this section to read as follows by the addition of italicized words:

> 7. (*a*) Where lead or lead-alloy coated wire is used, the continuity of the lead coating shall be determined in accordance with either Section 7 *or Section 8* of the Standard Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (A.S.T.M. Designation: B 189). *If a tin-coated wire is used, it shall conform to the Standard Specifications for Tinned Soft or Annealed Copper Wire for*

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[2] 1949 Book of A.S.T.M. Standards, Part 6.

Electrical Purposes (A.S.T.M. Designation: B 33)."

Table II, Thickness of Insulation and Alternating Test Voltage.—Include the first line, covering No. 18 to No. 16 wire, in the 0 to 600 v. class and omit the 0 to 300 v. classification. Delete Footnote (*b*) referring to these sizes.

Section 25.—Change the second sentence to read as follows by the addition of the italicized words: "The tape shall be treated on *at least* one side with an insulating compound of a nature not injurious to the wire insulation."

Section 31 (b).—In the second sentence of this section include a reference to Table VII, as well as to Table VIII or Table IX.

Section 37.—Change to read as follows by the addition of the italicized words: "The moisture-resistant properties of the braid covering on *single conductor* wires and cables, etc."

Tentative Methods of Chemical Analysis of Rubber Products (**D 297 - 43 T**).[2]—In this revision, as appended[3] to this report, these methods of analysis have been completely rewritten including: additional definitions, rearrangement into a more usable form, and changes to bring the methods into general agreement with recent revisions of Federal Specifications Board methods in the same field. While work still remains to be done and is in progress on improvement of existing methods and the addition of new ones, the present revision represents an important step forward.

Tentative Methods of Testing Rubber Hose (**D 380 - 49 T**).[2]—The following change is recommended in order to bring the immersion test conditions into conformity with present practice:

Section 25 (c).—Delete the fourth sentence and replace with the following: "When the test liquid is a fuel, the temperature during the immersion period shall be 70 to 90 F. except when arbitration is involved, in which case, a test atmosphere of 50 ± 4 per cent relative humidity at a temperature of 73.5 ± 2 F. shall be maintained. When the test liquid is an oil, the temperature shall be 212 ± 2 F. during the test."

Tentative Methods of Testing Rubber Insulated Wire and Cable (**D 470 - 49 T**).[2]—This revision provides for the determination of ozone concentration by either the present chemical analysis method or by direct measurement with an ozonometer. The latter method has the advantage of permitting continuous readings of the ozone concentration during test and is applicable for such use with ozone concentrations in the range specified in this method. The addition of the ozonometer method requires rearrangement of the sections dealing with the test.

Section 28.—Transpose the entire section on "Special Solutions Required" to new Section 31 as Item (*1*) in Paragraph (*a*). Renumber subsequent sections accordingly.

Section 29 (a).—Renumber as Section 28 (*a*).

Section 29 (b).—Renumber as Section 28 (*b*) and delete the last four sentences commencing "For determining ozone concentration, a sampling bottle etc."

Section 30.—Renumber as Section 29. Change Paragraph (*c*) to read as follows:

> (*c*) Specimens shall be bent without twisting at room temperature, but not less than 20 C., around a mandrel. The mandrel diameter shall be as follows:

Cable Outside Diameter	Mandrel Diameter
Less than ½ in.	4 × Cable O.D.
½ in. but less than ¾ in.	5 × Cable O.D.
¾ in. but less than 1¼ in.	6 × Cable O.D.
1¼ in. but less than 1¾ in.	8 × Cable O.D.
1¾ in. and above	10 × Cable O.D.

Section 31.—Renumber as Section 30 and correct reference in parentheses to Section 32 accordingly.

Sections 32 and 33.—Replace these sections with the following, renumbering as Section 31:

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[3] The revised method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 6.

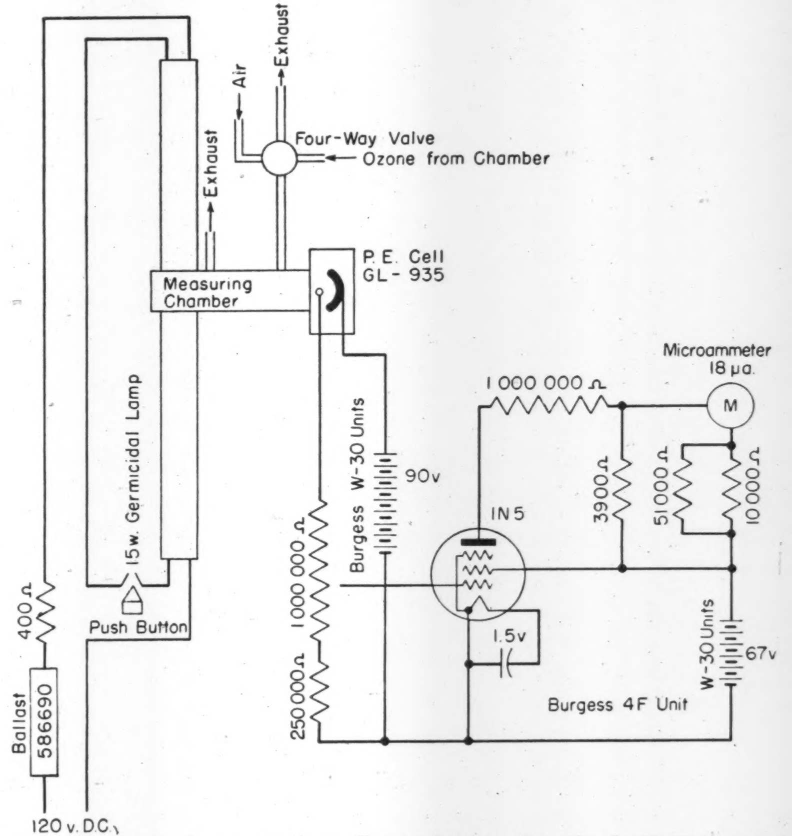
31. *Determination of Ozone Concentration.*—The ozone concentration shall be determined by one of the following approved methods:

(*a*) *Chemical Analysis:*

(*1*) Special Solutions Required.—(Insert text of present Section 28 (*a*), (*b*), (*c*), (*d*), and (*e*)).

(*2*) Collection of Sample.—(Insert text of present Section 32 (*a*)).

(*3*) Analysis of Sample.—(Insert text of present Section 32 (*b*)).


Exhaust
Air
Exhaust
Four-Way Valve
Ozone from Chamber
P. E. Cell
GL-935
Measuring Chamber
15w. Germicidal Lamp
Push Button
400 Ω
Ballast
586690
120 v. D.C.
1000 000 Ω
250 000 Ω
Burgess W-30 Units
90v
Microammeter
18 μa.
1 000 000 Ω
M
3900 Ω
51000 Ω
10 000 Ω
IN5
1.5v
W-30 Units
67v
Burgess 4F Unit

NOTE.—Resistor values in microammeter shunt will vary with the meter used and with the concentration scale desired.

FIG. 1.—Electronic Ozonometer.

(*4*) Calculations.—(Insert text of present Section 33 (*a*) and (*b*)).

(*b*) *Direct Measurement with an Ozonometer.*—This method is based on the absorption of 2537Å radiation by ozone. The equipment is composed of a source of 2537Å radiation and a photoelectric cell located on the opposite sides of a measuring cell through which the ozone atmosphere to be measured is passed. The amount of 2537Å radiation that is available for activating the photoelectric cell is dependent on the concentration of ozone in the measuring chamber. The current generated in the photoelectric cell is amplified sufficiently to be read directly on a sensitive milliameter. Figure 4 (the accompanying Fig. 1) shows a suggested schematic arrangement of this apparatus. The microammeter can be marked to read directly in per cent ozone. The calibration of this instrument should be made by comparison with results obtained with the chemical method (Paragraph (*a*)). The advantage in using this method is that after a calibration is obtained the ozone concentration is continuously readable on the microammeter without drawing a sample from the test chamber and thus upsetting equilibrium.

Tentative Methods of Testing Hard Rubber Products (**D 530 - 48 T**).[2]—This revision changes the tensile strength and elongation determination of hard rubber by specifying the rate of load application during test rather than the rate of jaw separation of the testing machine. Round-robin test programs which have been carried out by Subcommittee XXIII on Hard Rubber have shown that this change will somewhat improve the reproducibility of the tensile strength and elongation tests.

Section 7 (a).—Delete the third requirement, reading "The rate of travel of the power-actuated grip shall be 0.4 in. per min. and shall be uniform at all times" and replace with "The rate of travel by the power-actuated grip shall be such that the average rate of load application shall be not more than 3.1 nor less than 2.9 lb. per sec. when measured with a non-extensible steel bar locked in the grips."

Tentative Specifications for Insulated Wire and Cable: Polyvinyl Insulating Compound (**D 734 - 43 T**).[2]—This revision constitutes a minor change to conform to Specifications B 8.

Section 6, Permissible Variations in Diameter and Area.—Change the value of "1 per cent under" to "1 per cent under for solid conductors and 2 per cent under for stranded conductors" in the permissible variations specified in this section.

Tentative Specifications for Insulated Wire and Cable: Heat-Resisting Synthetic Rubber Compound (**D 754 - 46 a T**)[2] and *Tentative Specifications for Insulated Wire and Cable: Performance Synthetic Rubber Compound* (**D 755 - 46 a T**).[2]—These revisions are intended to bring these specifications into agreement with the basic provisions of Specifications D 27.

Table II, Thickness of Insulation and Alternating Test Voltage.—Include the first line containing No. 18 to No. 16 wire in the 0 to 600-v. class and omit the 0 to 300-v. classification. Delete footnote (*b*) and the reference to it in Table II. Add the following as NOTE 5 at the top of Table II: "NOTE 5.—Above 5000 v. a.c., ozone resistant insulation is recommended as prescribed in the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (A.S.T.M. Designation: D 574).

Tentative Method of Heat Aging of Vulcanized Natural or Synthetic Rubber by Test Tube Method (**D 865 - 48 T**).[2]—This revision consists of the addition of one sentence in Section 4 (*e*).

Section 4 (e).—Add the following sentence: "A check of the actual temperature within the test tube should be made by placing a thermometer in the tube with the bulb in the location ordinarily occupied by the specimens and the stem extending through a third hole drilled in the cork stopper.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in five standards and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Specifications for Insulated Wire and Cable: Performance Rubber Compound (**D 353 - 48**)[2] and *Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound* (**D 469 - 46**).[2]—This revision changes the rated circuit voltage classification of No. 18 to No. 16 conductors to bring these specifications into conformity with current practice.

Table II, Thickness of Insulation and Alternating Test Voltage.—Include the first line containing No. 18 to No. 16 wire in the 0 to 600-v. class and omit the 0 to 300-v. classification. Delete footnote *a* pertaining to sizes No. 16 and No. 18 and the reference to it in Table II.

Standard Method of Air Pressure Heat Test of Vulcanized Rubber (**D 454 - 48**).[2]

—This revision clarifies the provisions of the method relating to heating media and methods for measuring temperature.

Section 4 (b).—Change to read as follows:

(*b*) The operating temperature shall be 126.7 ± 1 C. (260 ± 1.8 F.) determined as described in Section 4 (*e*). The temperature shall be automatically controlled by means of thermostatic regulation.

Section 4 (c).—Change to read as follows:

(*c*) The source of heat is optional, but if located inside the aging chamber, shielding shall be provided so that direct radiation cannot reach the specimens. The temperature of the shield surfaces shall be within 1 C. of the air temperature.

Section 4 (d).—Reletter present paragraph as (*f*) and add the following as new Paragraph (*d*):

(*d*) The heating medium is optional. Steam, air, or liquid media may be used. If air is used, the heated air shall be thoroughly circulated by means of mechanical agitation, and baffles shall be used as required to prevent local overheating and dead spots. Oils or other combustible organic fluids may be hazardous at the elevated temperature required, but if their use is necessary they must have a flash point not lower than 200 C. For any one type of heat transfer medium, complete immersion of the pressure vessel in the heating medium is recommended for referee purposes in order to assure uniformity of temperature inside the vessel.

Section 4 (e).—Reletter the present paragraph as (*g*) and add the following as Paragraph (*e*):

(*e*) The temperature shall be automatically recorded throughout the test period. If the pressure chamber is completely immersed, the temperature may be taken as that of the heating medium. The sensitive element of the temperature-measuring device shall be close to the pressure chamber, but not touching it. If the pressure chamber is not completely immersed, the sensitive element shall be placed in a thermometer well extending into the pressure chamber. The thermometer well should be filled with oil or mercury to a depth sufficient to cover the sensitive element, in order to facilitate heat transfer. In any case, it is desirable to verify the recorded temperature, and the uniformity of temperature distribution at different points within the pressure chamber, by checking with a temperature-indicating device having its sensitive element directly exposed to the air within the pressure chamber.

Section 4 (f).—Reletter as Paragraph (*h*).

Section 4 (g).—Reletter as Paragraph (*i*) and revise by the deletion of the word in brackets:

(*i*) No copper or brass parts shall be exposed to the atmosphere [used] in the pressure chamber.

Section 4 (h).—Reletter as Paragraph (*j*) and revise by the addition of italicized words and deletion of those in brackets:

(*j*) The pressure chamber shall be equipped with a reliable safety valve *or rupture diaphragm* set for release at a pressure of not more than [200] *500* psi.

Note.—Add the following to the note at the end of Section 4:

If the same equipment is used for the air pressure heat test and the oxygen-pressure test (Method D 572), combustible heating media should not be used.

Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (**D 572 - 48**).[2]—This revision clarifies the provision relating .to heating media and methods for measuring temperature.

Section 4.—Delete the second sentence of the first paragraph, together with the figures to which it refers, and substitute the following: "Because of the superior temperature control and heat transfer, metal vessels completely immersed in a liquid medium are recommended for purposes of referee tests."

Section 4 (c).—Change to read as follows:

(*c*) The heating medium is optional. Water, air, or other fluids known to be safe in the presence of oxygen may be used. Water has an advantage because of its rapid heat transfer and noncombustible nature. If air is used, the heated air shall be thoroughly circulated by means of mechanical agitation, and baffles shall be used as required to prevent local overheating and dead spots. Oils or other combustible fluids are extremely hazardous in the presence of oxygen, and should not be used as heating media for this test.

Section 4 (*d*).—Delete the second sentence.

Section 4 (*e*).—Change to read as follows:

> (*e*) The temperature shall be automatically recorded throughout the test period. If the pressure chamber is completely immersed, the temperature may be taken as that of the heating medium. The sensitive element of the temperature-measuring device shall be close to the pressure chamber but not touching it. If the pressure chamber is not completely immersed in the heating medium, the sensitive element may be placed in a thermometer well extending into the pressure chamber. The thermometer well should be filled with water, oil, or mercury to a depth sufficient to cover the element, in order to facilitate heat transfer. If it is confirmed by actual check that the temperature of the oxygen within the chamber is the same as that of the heating medium, the temperature may be taken in the heating medium instead of in the thermometer well. If air is used as the heating medium, a check of the oven temperature shall be made by means of temperature-indicating devices placed in various parts of the oven to verify the uniformity of heating. In any case, it is desirable to verify the recorded temperature by checking with a temperature-indicating device having its sensitive element directly exposed to the oxygen within the pressure chamber.

Section 4 (*g*).—Change to read as follows by the addition of the italicized words: "(*g*) The pressure chamber shall be equipped with a reliable safety valve *or rupture diaphragm* set for release at 500 psi. pressure."

Section 4 (*h*).—Add new Paragraph (*h*) to read as follows: "(*h*) No copper or brass parts shall be exposed to the atmosphere in the pressure chamber."

Note 2.—Add the following to the note at the end of Section 4:

If the same equipment is used for the oxygen-pressure test and the air pressure heat test (Method D 454), care must be exercised to see that the themostatic controls are properly set, since the specimens may react very rapidly with oxygen at the temperature of the air-pressure heat test. Fluids which are acceptable as heating media for one test may be hazardous when used for the other test.

Section 8 (*b*).—Change to read as follows, by the addition of the italicized words and the omission of those in brackets: "The operating temperature shall be 70 ± 1 C. determined [by measuring the temperature of the heating medium which shall be considered the same as that of the pressure chamber] *as described in Section 4* (*e*)."

Standard Method of Testing Compressed Asbestos Sheet Packing (**D 733 – 49**).[2]—It is proposed to revise this method by deletion of Section 11. After careful study, Subcommittee VI on Packings has found that this test, in which bone dry samples are employed and no aging medium is given, is not used. In its place, consumers have used methods involving preconditioning under varying degrees of relative humidity and aging in air ovens for varying times and temperatures. The committee has found that under procedures for preconditioning specimens at high humidity and aging at low temperature all sheets corrode all metals and with preconditioning at moderate humidities and aging at elevated temperatures, no corrosion occurs on any sheet on any metal. The committee believes that under service conditions, no corrosion occurs and knows of no combination of preconditioning and aging procedures which will differentiate sheets in their action on metals. The committee further recommends that the note in Section 10 (*d*) reading "NOTE.—Thickness change is preferred to volume change as an index to the effect of liquid media as no accurate method is known for determining the latter." and published as a tentative revision of D 733, be incorporated into the standard.

ADOPTION OF TENTATIVE REVISION AS STANDARD

Standard Method of Testing Compressed Asbestos Sheet Packing (D 733 - 49).—The committee recommends that the present tentative revision of Section 10 (*d*) be adopted as standard and incorporated in this method.

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

Standard Methods of Sample Preparation for Physical Testing of Rubber Products (**D 15 - 41**),[2] *Standard Methods of Testing Flat Rubber Belting* (**D 378 - 41**).[2]—Extensive revisions of these standard methods are in progress, and it is expected that they will be presented to the Society through the Administrative Committee on Standards prior to the next Annual Meeting. To facilitate this work, the committee requests that these standards be reverted to tentative status.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that all tentatives under its jurisdiction other than those referred to in this report be continued as tentative. Work is under way in the committee on most of these, and the results will be presented to the Society as it is completed.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Organization of a new subcommittee to carry on standardization work on crude natural rubber has progressed through the preliminary stages, following its authorization at the last Annual Meeting of the committee, and this new Subcommittee XII, under the chairmanship of N. Bekkedahl, held its first meeting April 19, 1950, at Detroit, Mich. Eighteen prominent rubber technologists, representing a wide cross-section of the industry, were in attendance and enthusiastic plans were developed covering work to be undertaken. Prior to the recent World War, a similar crude rubber committee functioned in the Rubber Division of the American Chemical Society and accomplished much good work in the development and the standardization of test formulations and quality standards for this basic raw material. During the war this committee became inactive. Last year several rubber consumers requested Committee D-11 to undertake work in this field. When it was found that the rubber division committee would not be reactivated, it was decided to organize the new subcommittee. Subcommittee XII has for its present objective the improvement and development of methods of physical and chemical testing of crude natural rubber, possibly including investigation of sampling procedures. This work is badly needed since crude natural rubber has again become available and because of wide variations being encountered in curing rate and processing characteristics, as well as presence of substantial quantities of foreign material, all of which contrasts strongly with the uniformity which has now been secured in the manufacture of synthetic materials.

Organization of the new Subcommittee, which has been authorized, on the Application of Statistical Quality Control Methods to Rubber Products is still delayed, pending the securing of a chairman and the appointment of suitable personnel. The committee has also authorized an editorial subcommittee, which is in process of organization.

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[4] The letter ballot vote on these recommendations was favorable: the results of the vote are on record at ASTM Headquarters.

C. P. Morgan has been appointed chairman of Subcommittee XXIII on Hard Rubber, replacing H. J. Flikkie who has resigned because of a change in his activities. The title of Subcommittee XXI has been changed from "Tests of Liquid Rubber Products" to "Tests of Rubber Cements and Related Materials." Originally, it was contemplated that work on rubber latices would be under the jurisdiction of this subcommittee, but with the formation of Subcommittee VII, it is believed the new title more accurately describes the field of activity of Subcommittee XXI.

The activities of the various subcommittees are so numerous that detailed consideration is beyond the scope of this report. It is expected, however, that Subcommittee IV will complete a new specification for electrical gloves in the near future. Subcommittee VI expects soon to recommend a method for measuring the compressibility and recovery of packings. Subcommittee X is progressing rapidly on the revision of Methods D 15, Standard Methods for Sample Preparation for Physical Testng of Rubber Products. A new Test Method for Accelerated Ozone Cracking of Vulcanized Rubber is under consideration in draft form in Subcommittee XV. Several of the subcommittees have extensive cooperative test programs under way, of which perhaps the most extensive is that of Subcommittee XV on the correlation of oven and shelf aging, in which results have already been obtained covering a period of two years. Subcommittee XXI is expected soon to complete proposed Tentative Methods of Test for Rubber Brake Bonding Materials. This brief summary merely exemplifies the many projects which are under way in the various subcommittees, demonstrating the fact that Committee D-11 is fully aware of its responsibility in the field of rubber standardization.

This report has been submitted to letter ballot of the committee, which consists of 198 members; 131 members returned their ballots, of whom 113 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

SIMON COLLIER,
Chairman.

ARTHUR W. CARPENTER,
Secretary.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS[1]

Committee D-12 on Soaps and Other Detergents held one meeting during the year, on March 21 and 22 in New York, N.Y.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Jay C. Harris.
Vice Chairman, Frederick Krassner.
Secretary, Harold R. Suter
Advisory Committee: J. C. Harris, Frederick Krassner, H. R. Suter, J. A. Woodhead, W. G. Morse, G. P. Fulton, W. H. Koch, V. C. Mehlenbacher, R. E. Hauber.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

Committee D-12 recommends that the following proposed methods of test be published as information, as appended hereto:

Proposed Method of Test for pH of Aqueous Solutions of Soaps and Detergents,
Proposed Method of Test for Foaming Properties of Surface-Active Agents.

NEW TENTATIVES

The committee recommends that the following specifications be accepted for publications as tentative, as appended hereto:[2]

Tentative Specifications for:

Solid Soap for Low-Temperature Washing (Low and Medium Titer), and
Chip or Granular Soap for Low-Temperature Washing (Low and Medium Titer).

ADOPTION OF TENTATIVE AS STANDARD

Tentative Specifications for Borax (**D 929 - 47 T**).[3]—The committee recommends that these specifications be approved for reference to letter ballot of the Society for adoption as standard.

EDITORIAL CHANGE IN STANDARD

Standard Specifications for Alkaline Soap Powder (**D 534 - 42**).—The committee recommends that this standard be editorially revised by eliminating the waiver-purchase clause to conform with all the other soap specifications.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends that the Tentative Specifications for Sodium Bicarbonate (D 928 - 47 T) be retained as tentative without revision.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revision in the Standard Definition of Terms Relating to Soaps and Other Detergents (D 459 - 48) and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Change the definition for "Alkaline Detergent" to read as follows:

Alkaline Detergent.—A water-soluble inorganic alkali or alkaline salt having detergent properties but containing no soap or synthetic detergent.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The new tentative specifications were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[3] 1949 Book, of A.S.T.M. Standards, Part 5.

The recommendations appearing in this report have been submitted to letter ballot of the Committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee T-1 on Soap Analysis (E. W. Blank).—This subcommittee, which functions as a joint committee with the American Oil Chemists' Society, reports that cooperative tests in the determination of borax in soaps and in combination with synthetic detergent are to be undertaken. A further cooperative effort will involve the study of the water-soluble determination in soap.

Subcommittee T-4 on Analysis of Alkaline Detergents (W. A. Koch, chairman). —This subcommittee expects to complete a method for analysis of sodium bicarbonate within the coming year. Other new methods for analysis will depend upon the needs developed through the investigations of Subcommittee S-4.

Subcommittee T-5 on Physical Testing (J. A. Woodhead, chairman).—The subcommittee recommends that the following be published as information only, as mentioned earlier in this report: Proposed Method for the Determination of pH of Aqueous Solutions of Soaps and Detergents, and the Proposed Method of Test for Foaming Properties of Surface Active Agents. These methods were prepared by H. R. Suter and C. A. Cohen, respectively. A Task Group has been appointed to present comparative data on wetting test methods and work is underway on the preparation of methods for testing surface and interfacial tension.

Subcommittee T-6 on Metal Cleaning (J. C. Harris, Chairman).—This group expects to complete during the coming year methods for Immersion Corrosion Test for Soak Tank Metal Cleaners, Rinsing Characteristics, Buffering Capacity, and Emulsification.

Subcommittee S-1 on Specifications for Soaps (Fred Krassner, chairman).—The subcommittee recommended the publication as tentative of specifications for Solid Soap (Low and Medium Titer) and Chip or Granular Soap (Low or Medium Titer), as mentioned earlier in this report, and the editorial change in specifications D 534, Soap Powder, as mentioned earlier in the report.

Subcommittee S-3 and T-3 on Dry Cleaning (G. P. Fulton, chairman) has carried out flash-point determinations for 28 commercial dry cleaning soaps and detergents using the Pensky-Martens Tester and the Tag Closed Tester. A cooperative test for determination of reproducibility between laboratories is to be completed this year. A comparison of the xylol distillation and the Karl Fischer methods of water determinations indicated the superiority of the latter, and a method based upon it is to be tried out.

Subcommittee G-1, Advisory Committee. —A new subcommittee, G-5 on Program Planning, was established. The function of the new group is to provide competent speakers for the subcommittee meetings, as required, and for the general session.

The subcommittee has approved a change in the names of Subcommittees S-4 and T-4 from "Special Detergents" to "Alkaline Detergents."

The need for greater representation from consumer and general interest groups has been emphasized.

Subcommittee G-2 on Nomenclature and Definitions (W. G. Morse, chairman).—A change was recommended in the definition of an alkaline detergent. Several other definitions are required as a result of requests from the various subcommittees, and these are under discussion.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

This report has been submitted to letter ballot of the committee, which consists of 104 members; 81 members returned their ballots, of whom 73 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. W. SMITHER,
Chairman.

J. C. HARRIS,
Secretary.

APPENDIX I

PROPOSED METHOD OF TEST FOR pH OF AQUEOUS SOLUTIONS OF SOAPS AND DETERGENTS[1,2]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method covers procedures for the preparation of aqueous solutions of soaps and detergents and for the determination of their pH.

Sampling

2. The material shall be sampled in accordance with the Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (A.S.T.M. Designation: D 460),[3] or the Standard Methods of Sampling and Chemical Analysis of Alkaline Detergents (A.S.T.M. Designation: D 501),[3] whichever is applicable.

Reagent

3. *Distilled Water.*—Distilled water shall be boiled thoroughly, or purged with CO_2-free air, to remove CO_2 and shall be protected with soda-lime or soda-asbestos (Ascarite) while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 25 C. The residue on evaporation, when heated at 105 C. for 1 hr., shall not be more than 0.5 mg. per l.

Procedure

4. (*a*) *Preparation of Solutions.*—Weigh 1 ± 0.001 g. of the soap or detergent and transfer to a 1-liter volumetric flask. Partially fill the flask with distilled water and agitate until the sample is completely dissolved. Adjust the temperature of the solution and the distilled water to 25 ± 0.5 C. and fill to the calibration mark with distilled water. Stopper the flask, mix thoroughly, and allow the solution to stand at a temperature of 25 C. for 2 hr. prior to measuring the pH.

(*b*) *Determination of pH.*—Measure the pH of the solution as directed in the Tentative Method for Determination of the pH of Aqueous Solutions with the Glass Electrode (A.S.T.M. Designation: E 70).[3] Use the "low sodium-error" glass electrodes for solutions having pH values higher than 9. Use type I or type II meters.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Other Detergents.

[2] Published as information, June, 1950.

[3] 1949 Book of A.S.T.M. Standards, Part 5.

APPENDIX II

PROPOSED METHOD OF TEST FOR FOAMING PROPERTIES OF SURFACE-ACTIVE AGENTS[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test is intended for determining the foaming properties of surface-active agents, as defined in the Standard Definitions of Terms Relating to Soaps and Other Detergents (A.S.T.M. Designation: D 459).[2] The method is applicable under limited and controlled conditions, but does not necessarily yield information correlating with specific end uses.

Apparatus

2. (*a*) *Pipet*.—The pipet (Fig. 1) shall be constructed from standard-wall, chemically resistant glass tubing having the following dimensions: for the bulb, 45 ± 1.5 mm. outside diameter; for the lower stem, 7 ± 0.5 mm. outside diameter. The upper stem shall be constructed to contain a solid-stopper, straight bore, No. 2, standard-taper stopcock having a 2-mm. bore and stems 8 mm. in outside diameter. Both the upper and lower seals of the bulb to the stems shall be hemispherical in shape. The lower stem shall be 60 ± 2 mm. in length from the point of attachment to the bulb and shall contain an orifice sealed into the lower end. The orifice shall be constructed from precision bore tubing having an inside diameter of 2.9 ± 0.02 mm. and a length of 10 ± .05 mm., with both ends ground square. The orifice shall have an outside diameter so as to fit snugly into the lower stem and form a secure seal to the stem when heated with a sharp pointed flame in the blow torch. The pipet shall be calibrated to contain 200 ± 0.2 ml. at 20 C. The calibration mark shall be on the upper stem at least 15 mm. below the barrel of the stopcock and shall completely encircle the stem.

(*b*) *Receiver*.—The receiver (Fig. 2) shall be constructed from standard-wall, chemically resistant glass tubing having an internal diameter of 50 ± 0.8 mm., with one end constricted and sealed to a straight-bore, solid-plug, standard-taper No. 6 stopcock having a 6-mm. bore and 12-mm. stems. The receiver shall have three calibration marks which shall completely encircle the tube. The first mark shall be at the 50-ml. point, shall be measured with the stopcock closed, and shall not be on any curved portion of the constriction. The second mark shall be at the 250-ml. point, and the third mark at a distance of 90 ± 0.5 mm. above the 50-ml. mark. The receiver tube shall be mounted in a standard-wall tubular water jacket, having an external diameter of not less than 70 mm., fitted with inlet and outlet connections. The jacket may be attached to the receiver with rubber stoppers or may be sealed at the top and bottom. The seal at the bottom shall be as close to the barrel of the stopcock as practicable. The assembled receiver and jacket shall be mounted securely in a plumb position and the jacket connected to a source of water thermostatically maintained at 120 ± 1 F. for circulating through the jacket. At the top of the receiver there shall be a platform, flush with the top of the assembly, having a metal plate in which is drilled three indexing holes circumferentially placed around the receiver and having an angular displacement of 120 F. from each other. A clamp which may be securely attached to the upper part of the pipet, shall fit into the holes. The clamp shall have three leveling screws and lock nuts and when properly mounted shall exactly center the pipet in the receiver and bring the lower tip of the pipet level with the upper calibration mark on the receiver. A meter stick shall be fastened to the side or behind the receiver with the zero point level with the 250-ml. calibration point on the receiver.

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-12 on Soaps and Other Detergents. Published as information, June, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 5.

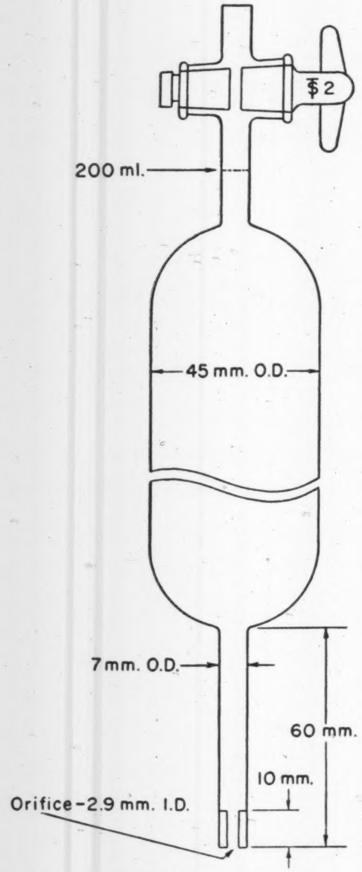

FIG. 1.—Foam Pipet.

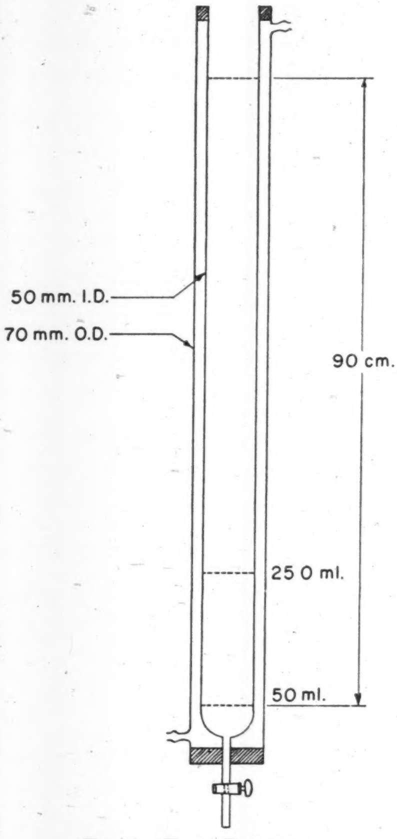

FIG. 2.—Foam Receiver.

Test Solution

3. Distilled water, or water of various degrees of hardness, may be used for this test. Preheat the water used for pre-

paring the solution and add slowly, while stirring vigorously, an amount of the surface-active agent that will produce the desired concentration. Continue stirring in such a manner as to avoid excessive foam formation, until solution of the surface-active agent is complete. Age the solution at a temperature of 120 F. for a total period of 30 min., counting the time when the surface-active agent is first added to the water.

Procedure

4. While the surface-active solution is aging, circulate water at 120 F. through the water jacket of the receiver so as to bring it to the proper temperature. Rinse down the walls of the receiver with distilled water and, as an indication of cleanliness, observe whether the water drains down the walls in an unbroken film. At the completion of the aging period close the stopcock at the bottom of the receiver. Rinse the walls of the receiver with 50 ml. of the solution, using a pipet, and, after draining to the bottom of the receiver, adjust the stopcock so that the level of the solution in the receiver is exactly at the 50-ml. mark. Fill the pipet with the solution to the 200-ml. mark, using a slight suction for the purpose. Immediately place it in position at the top of the receiver and open the stopcock. When all of the solution has run out of the pipet, start a stop watch, take a reading of the foam height and take a second reading at the end of 5 min. Take the reading by measuring the foam production at the top of the foam column at the highest average height to which the rim of the foam has reached. This height is proportional to the volume of air remaining in the foam.

Report

5. In reporting results by this test, state the concentration in grams per liter, the temperature of the test, the degree of hardness of the water, and the initial foam height reading. It is desirable to conduct this test at a number of concentrations, the lowest of which shall be chosen to show a foam height which shall be no higher than 20 per cent of the foam height shown at the highest concentration.

REPORT OF COMMITTEE D-13

ON

TEXTILE MATERIALS[1]

Committee D-13 on Textile Materials held two 3-day meetings during the year. The fall meeting was in Philadelphia, Pa. on October 19 to 21, 1949, and there was a registered attendance of 195 members and guests; the spring meeting was in New York, N. Y. on March 15 to 17, 1950, with a total attendance of 213. Fifteen subcommittees and sections held sessions at both the fall and spring meetings. Two other subcommittees found it more convenient to meet at other times and places. Administrative guidance of the committee required two meetings of the Advisory Committee and one of the Officers Committee.

The papers session at the fall meeting was devoted to the subject of modern techniques in microscopy and their application in textile research. The following papers of an informative character were presented: "A New Technique for Cutting Very Thin Sections and Its Application to the Electron Microscopy of Fibers," by S. B. Newman, National Bureau of Standards; "Modern Microscopy of Films and Fibers," by F. F. Morehead, American Viscose Corp.; and "Some Applications of Modern Microscopy to the Study of Chemical Phenomena and in the Dyeing and Printing of Textiles," by G. L. Royer, American Cyanamid Co. The first two papers were published in the January, 1950, issue of the ASTM Bulletin and the third appeared in the April, 1950, issue. The general subject selected for the spring meeting was "The Dimensional Stability and Shrink-Proofing of Textiles." The subdivisions of the topic and the authors selected for each were as follows: "Cotton Textiles," by Edward C. Pfeffer, Jr., Cluett Peabody and Company, Inc.; "Rayon Textiles," by J. A. Woodruff, American Viscose Corp., and "Woolen Fabrics," by Werner von Bergen and Claude Clutz, Forstmann Woolen Co. It is proposed to publish these three papers in the July issue of the ASTM Bulletin.[2]

Some important changes have been made in the organization of Committee D-13 during the year. The two subcommittees which dealt chiefly with ultimate consumer textiles, namely, Subcommittee A-6 on Household and Garment Fabrics and Subcommittee A-12 on Hosiery, have been discontinued. In their place has been set up a new Subcommittee A-7 on Ultimate Consumer Textile Products, with A. F. Tesi as chairman and E. N. Ditton as secretary. These changes now clearly designate where work on ultimate consumer textiles will be handled in Committee D-13. It is contemplated that appropriate subdivisions, such as sections or task groups, will be organized to handle specific projects as the need arises. Furthermore, it is planned to inform other national associations of the existence of this subcommittee as a place where test methods for consumer textiles may be developed.

The following changes in subcommittee and section chairmanships occurred during the year and have been approved by the Advisory Committee: A. F.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] Published in the ASTM Bulletin, No. 167, July, pp. 74 to 87.

Tesi, vice Laura E. Pratt, as chairman of A-6; F. A. Bartlett, vice J. F. Wooten, as chairman of Subcommittee A-8, Section II; G. B. Krumholz, vice K. N. Mathes, as chairman of Subcommittee A-8, Section III; E. A. Leonard, vice S. L. Peebles, as chairman of Subcommittee A-11; W. M. Scott, vice W. D. Appel, as chairman of Subcommittee B-1.

Upon the recommendation of the Advisory Committee, the Society has appointed A. F. Tesi as its representative on ASA Sectional Committee L-22 on Rayon Finished Fabrics. A. G. Scroggie and Frederic Bonnet were appointed as first and second alternates, respectively. R. H. Brown, a long-time and valued member of Committee D-13, is a nominee for election to the Board of Directors of the Society for the ensuing term of three years.

At the fall meeting a testimonial luncheon was held, under the sponsorship of Subcommittee A-3 on Wool, to honor A. G. Ashcroft and Werner von Bergen. Each was presented an appropriately worded scroll, which recorded the long service they had rendered and expressed well-earned recognition of the character and importance of their contribution to the standardization programs in the carpet and wool industries.

The spring meeting was made memorable by the ceremony accompanying the first award of the Harold DeWitt Smith Memorial Medal to Herbert F. Schiefer of the National Bureau of Standards. He was presented to the audience by Richard T. Kropf, who spoke of the personal qualifications of the medalist, and by William D. Appel, who described his scientific work and achievements. The gold medal and a suitable citation in scroll form, both provided through the generosity of Fabric Research Laboratories, Inc., were presented by H. J. Ball, chairman of the Award Committee.

To mark the completion of twenty years of service as chairman of Committee D-13, a testimonial luncheon was tendered to the retiring chairman at the spring meeting. As master of ceremonies, secretary W. H. Whitcomb, told of his close relationship with the chairman throughout the twenty-year period. First Vice-Chairman G. E. Hopkins reviewed the progress which Committee D-13 had made during the chairman's tenure of office, listed the other positions in which the chairman had served the Society, and closed his remarks with the presentation of a beautifully designed and worded scroll. Second Vice-Chairman Frederic Bonnet followed with the presentation of a gift of money, generously contributed by the members of the committee. The chairman here takes the opportunity to repeat those parts of his response in which he expressed his sincere thanks to all those who by their efforts and contributions made the testimonial luncheon a happy memory, his belief that the success and prestige to which Committee D-13 has attained is due in large part to the magnificent and wholehearted cooperation of all those who have served as officers and members of the Advisory Committee, and his prediction that the coming twenty years hold the potential for as great progress as the past twenty.

It is with deep regret and sadness that we record the death of C. B. Finckel. He was one of the original organizers of Committee D-13 in 1914, and had been an honorary member since 1947. He will be remembered for the many years of faithful conscientious service which he gave to advance the interests of Committee D-13, and for that genial happy disposition which made him such a likeable personality at all our gatherings, both technical and social.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, W. D. Appel.
First Vice-Chairman, Frederic Bonnet.
Second Vice-Chairman, S. J. Hayes.
Secretary, W. H. Whitcomb.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-13 presented to the Society through the Administrative Committee on Standards a tentative revision of the Standard Method of Test for Hard Scoured Wool in Wool in the Grease (D 584 - 47). This recommendation was accepted by the Standards Committee on September 22, 1949, and the tentative revision appears in the 1949 Book of A.S.T.M. Standards, Part 5.

RECOMMENDATIONS AFFECTING STANDARDS

Committee D-13 is presenting for publication six new tentatives and is recommending the revision of eleven tentatives, tentative revisions in three standards, adoption as standard of several definitions and one tentative specification, immediate revision of two standards, the adoption as standard of the current tentative revisions of three standards, the withdrawal of two tentatives, and editorial changes in three tentatives.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.

All other tentatives not specifically referred to are being actively studied by the respective sponsoring subcommittees.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Besides the activities indicated by the recommendations appearing in the Appendix, the following summary covers other matters that are under consideration.

Subcommittee A-1 on Cotton and Its Products (B. L. Whittier, chairman):

Secion I on Cotton (R. L. Lee, Jr., chairman) is continuing its study of the methods of evaluating fineness. It is also planning interlaboratory checks of the methods of testing the cotton fiber characteristics.

Section II on Cotton Yarns and Threads (W. R. Marsden, chairman) is continuing its work on a revision of the Standard Methods of Testing and Tolerances for Cotton Sewing Thread (D 204 - 42).

Subcommittee A-2 on Man-Made Organic Base Fibers and Their Products (A. M. Tenney, Chairman):

Section I on Filaments, Monofilaments, and Filament Yarns (J. K. Frederick, Jr., chairman) is continuing its studies of methods of testing spun and continuous filament man-made organic base yarns.

Subcommittee A-3 on Wool and Its Products (Werner von Bergen, chairman):

Section I on Wool (O. P. Beckwith, chairman) is continuing its survey of wool testing, determination of moisture and fineness. A wool grading stage micrometer has been developed. Methods for the determination of quantity of wool in defective or burry wools and tensile strength of wool are being studied.

Section III on Woolen and Worsted Yarns (H. R. Anderson, chairman) is working on revisions of the Specifications and Methods of Test for Woolen Yarns (D 403 - 48 T) and for Worsted Yarns (D 404 - 48 T). Specifications for twist are being studied.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee A-4 on Asbestos and Its Textile Products (F. S. Mapes, chairman) is considering the adoption of 50 per cent relative humidity as standard for testing asbestos textiles. It is endeavoring to increase interest in asbestos materials in other than the electrical field.

Subcommittee A-5 on Bast and Leaf Fibers and Their Products (S. J. Hayes, chairman) is continuing its studies of test methods for twine. Cut and loop breaking strength are being studied in place of knot breaking.

Subcommittee A-8 on Glass Fiber and Its Products (F. A. Mennerich, chairman) is continuing its study of abrasion tests on fabrics and is studying test methods for colorfastness of glass fabrics to washing.

Subcommittee A-9 on Tire Cord and Fabrics (F. C. Kennedy, chairman) is continuing its work on revisions and consolidation of the Tests and Tolerances for Cotton Tire Cord (D 179 - 46 T) and for Rayon Tire Cord (D 885 - 46 T). A study is being made of air-actuated clamps.

Subcommittee A-10 on Felt (W. H. Lehmberg, chairman) is studying stiffness testers and various methods of measuring thickness of cut parts.

Subcommittee A-11 on Pile Fabrics (S. L. Peebles, chairman) is studying the Schiefer abrader and methods of test for flame resistance, ravel resistance, and light fastness.

Subcommittee A-14 on Bonded Fabrics (G. B. Harvey, chairman) is continuing its studies of air permeability, water absorbency, laundering and dry cleaning, tear resistance, abrasion resistance, flexural rigidity, and fire resistance.

Subcommittee B-1 on Test Methods (W. D. Appel, chairman) is developing specifications for abrasion machines and test methods for shrinkage of knit fabrics, fraying of fabrics, evenness of yarns, elastic fabrics, crease resistance, method of analysis of mixtures containing nylon, fluidity of cellulose dispersions, mildew resistance, and measurement of hand of fabrics.

Subcommittee B-2 on Nomenclature and Definitions (A. G. Scroggie, chairman) is continuing its study of certain definitions with a view to bringing about better agreement between A.S.T.M. and British definitions.

Subcommittee B-3 on Atmospheric Conditions and Regain (R. H. Brown, chairman) is continuing its studies on drying oven specifications; methods of determining moisture regain by oven drying; regain hysteresis and methods of preconditioning test samples; rate of conditioning; instruments for measuring regain and relative humidity.

This report has been submitted to letter ballot of the committee, which consists of 348 members; 168 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. J. BALL,
Chairman.

W. H. WHITCOMB,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials, which are referred to earlier in this report. These standards and tentatives appear in their present form in the 1949 Book of A.S.T.M. Standards, Part 5.

NEW TENTATIVES

Committee D-13 recommends the following six new tentatives for publication as tentative, as appended hereto.[1]

Tentative Method of Test for Vegetable Matter in Scoured Wool, prepared by Section I of Subcommittee A-3,

Tentative Method of Test for Magnetic Rating of Asbestos Used for Electrical Purposes, prepared by Subcommittee A-4, as published as information in Appendix X of the 1949 Compilation of "A.S.T.M. Standards on Textile Materials."

Tentative Specifications for Mechanical Sheet Felt, prepared by Subcommittee A-10,

Tentative Method of Test for Resistance of Pile Floor Coverings to Insect Pest Damage, prepared by Subcommittee A-11,

Tentative Method of Test for Snag Resistance of Hosiery, prepared by Subcommittee A-13,

Tentative Methods of Testing Bonded Fabrics, prepared by Subcommittee A-14.

REVISIONS OF TENTATIVES

Tentative Definitions of Terms Relating to Textile Materials (D 123 – 49 T):

The committee recommends that this tentative be revised by the addition of definitions of the following terms: bleaching; carding; extensibility; deformation; deformation, immediate elastic; deformation, delayed or creep; creep, primary; creep, secondary; yarn, Kraft; hair; and noil. These new definitions appear in the proposed revision of D 123 appended hereto.[2]

Tentative Method of Test for Fastness of Color Textiles to Light (D 506 – 45 T):

Revisions in this method to include an improved artificial light test, submitted by Subcommittee B-1, are recommended as appended hereto.[3]

Tentative Methods of Test for Resistance of Textile Fabrics to Water (D 583 – 40 T):

These improved methods prepared by Subcommittee B-1 are recommended as appended hereto.[3]

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[1] The new tentatives were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[2] The revised Definitions were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[3] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

Tentative Methods of Test for Fineness of Wool (D 419 - 47 T), and

Tentative Specifications and Methods of Test for Fineness of Wool Tops (D 472 - 47 T):

Revisions for the improvement and simplification of these methods were prepared by Section I of Subcommittee A-3 and are recommended as appended hereto.[3]

Tentative Method of Test for Determination of Small Amounts of Copper, Manganese and Nickel in Textiles (D 377 - 47 T):

Revisions in the methods for copper and manganese and withdrawal of the method for determining nickel were prepared by Subcommittee B-1 and are recommended as appended hereto.[3]

Tentative Specifications and Methods of Test for Asbestos Yarns (D 299 - 49 T):

The following revisions, to provide an optional sampling basis in case of retests, prepared by Subcommittee A-4, are recommended by Committee D-13:

Section 9.—In the fifth line of Paragraph (*a*) place a comma after the word "random" and insert the following words: "preferably from more than two cases."

Add the following as a new Paragraph (*c*):

(*c*) Optional with the purchaser, if the values as determined in accordance with Paragraph (*b*) fail to fall within the specified limits the lot shall be subdivided into units of approximately 200 lb. These shall be sampled and tested for conformance in accordance with Paragraphs (*a*) and (*b*).

Tentative Specifications and Methods of Test for Asbestos Lap (D 1061 - 49 T):

The following revision in the sampling procedure prepared by Subcommittee A-4, is recommended by Committee D-13:

Section 7.—Change to read as follows:

7. *Sampling.*—(*a*) Each shipment shall be divided into lots of from 800 to 1000 lb. From each lot or fraction thereof, five sample cones shall be taken at random and the tests prescribed shall be made on specimens removed from each package selected.

(*b*) If tests from any lot fail to fall within the specified limits, two additional sets of five sample cones shall be taken from other parts of the lot and tested as before. Acceptance shall be on the basis of these two sets of five sample cones being within the required limits.

(*c*) Optional with the purchaser, if the values determined in accordance with Paragraph (*b*) fail to fall within the specified limits, the lot shall be subdivided into units of approximately 200 lb. These shall be sampled and tested for conformance in accordance with Paragraphs (*a*) and (*b*).

Tentative Methods of Testing and Tolerances for Continuous Filament Rayon and Estron Yarns (D 258 - 48 T):

The following revisions to include the definitions of "Acetate" and "Acetate, Modified", prepared by Subcommittee A-2, are recommended by Committee D-13:

Section 2.—In Paragraph (*a*) number the present note as Note 1 and add as Note 2 the following:

NOTE 2.—Regenerated cellulose fibers made by the complete saponification of cellulose esters are rayons.

Add as new Paragraphs (*d*) and (*e*) the following definitions of "Acetate" and "Acetate, Modified," relettering the subsequent paragraphs accordingly:

(*d*) *Acetate, a* and *n.*—A specific term used for man-made fibers, monofilaments and continuous filament yarns composed of acetylated cellulose, with or without lesser amounts of nonfiber-forming material.

(*e*) *Acetate, Modified a* and *n.*—Acetate products composed principally of cellulose acetate and containing amounts of other fiber-forming material.

Add as Footnote 4 the following and

include reference to the footnote in the heading "definitions" preceding Section 2:

[4] The Federal Trade Commission's Fair Trade Practice Rules for the Rayon industry, issued October 26, 1937, include a definition of rayon and directions regarding its use. Any use of the above definitions should be made with full cognizance of the Federal Trade Commission Rules.

Tentative Methods of Testing and Tolerances for Glass Yarn (D 578 - 49 T):

The following new procedure for measuring thickness of glass cord was prepared by Subcommittee A-8:

New Section.—Add the following as a new Section 12, renumbering the present Section 12 as 13:

12. Cord Thickness: (*a*) *Apparatus.*—The gage used for the measurement of thickness of cords shall conform to the requirements prescribed in Section 11 of the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76).

(*b*) *Test Specimens.*—Test specimens shall be taken at least 2 yd. apart. Ten sets of test specimens are required for each determination. The number of test specimens per set shall be in accordance with the following table:

Cord Diameter, in.	Specimens, per set
$\frac{3}{32}$ and under	4
Over $\frac{3}{32}$ to $\frac{1}{8}$, incl.	3
Over $\frac{1}{8}$ to $\frac{1}{4}$, incl.	2
Over $\frac{1}{4}$	1

(*c*) *Procedure.*—The specified number of cords (Paragraph (*b*)) shall be laid side by side on the anvil of the gage in a relaxed condition and sufficiently close together to be contacted simultaneously by the presser foot. Care shall be taken in handling the cord to avoid distortion of twist and construction.

(*d*) *Report.*—The average of the ten tests shall be taken as the thickness of the cord.

TENTATIVE REVISIONS OF STANDARDS

Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 - 49):

On the recommendation of Subcommittee A-4 the following proposed changes are submitted as a tentative revision of this standard:

Section 2 (*d*).—Replace Paragraph (*d*) by the following:

(*d*) *Number* (*Cut*).—A number hyphenated with the word "cut" indicates the number of 100 yd. units in 1 lb. of a standard asbestos roving, and designates the cut-number of that roving.

Section 6.—Revise this section on sampling to read as indicated above for Section 7 in D 1061.

Section 8 (*a*).—In the first sentence change "six" to read "five."

Section 8 (*b*).—In the first sentence change "six strands" to read "five strands." Also change "24" in the first and second sentences, and in the formula and legend to read "20."

Standard Methods of Testing Rayon and Estron Staple (D 540 - 44):

On the recommendation of Section III of Subcommittee A-2 the following proposed changes are submitted as a tentative revision of this standard:

Section 2.—Add as new Paragraphs (*e*) and (*f*) the following definitions of "Acetate Staple" and "Acetate Staple, modified," relettering the subsequent paragraphs accordingly:

(*e*) *Acetate Staple.*—A specific term used for fibers composed of acetylated cellulose, with or without lesser amounts of nonfiber-forming material.

(*f*) *Acetate Staple, Modified.*—Acetate fibers composed principally of cellulose acetate and containing amounts of other fiber-forming material.

Add as Footnote 3 the following and include reference to the footnote in the heading "definitions" preceding Section 2:

[3] The Federal Trade Commission's Fair Trade Practice Rules for the rayon industry, issued October 26, 1937, include a definition of rayon and directions regarding its use. Any use of the above definitions should be made with full cognizance of the Federal Trade Commission Rules.

Standard Methods of Testing Felt (D 461 – 49):

On the recommendation of Subcommittee A-10 the following proposed change is submitted as a tentative revision of this standard:

Section 13 (*a*).—Change to read as follows:

> *13.* (*a*) *Sample.*—Prepare a sample of approximately 8 g. for chemical analysis by taking approximately equal quantities from three test specimens cut from the felt in accordance with Section 3. The portions so taken shall be cut into pieces approximately $\frac{1}{8}$ in. square.

ADOPTION OF TENTATIVES AS STANDARD

Tentative Specifications for Woven Asbestos Cloth (D 677 – 46 T):

The committee recommends that the above specifications be approved for reference to letter ballot of the Society for adoption as standard with editorial revisions in the table in Section 9 to make it agree with Table I in Section 4 (*a*) of D 299 – 49 T.

Tentative Definitions of Terms Relating to Textile Materials (D 123 – 49 T):

The committee recommends that the following definitions now published under D 123 – 49 T be approved for reference to letter ballot of the Society for adoption as standard: Monofilament; permeability; porosity; rayon; rayon, modified; ribbon, slit and cut; ribbon, woven; sley; tape, slit and cut; tape, woven; webbing, elastic; webbing, nonelastic; woolen; and yarn number.

It is also recommended that the following six terms now appearing in Tentative D 123 be adopted and included in the glossary which constitutes Appendix I of Standard D 123 – 49: corduroy, frieze, plush, velour, velvet, and velveteen.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

Committee D-13 recommends that the following standards be revised as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Definitions of Terms Relating to Textile Materials (D 123 – 49):

Committee D-13 recommends for immediate adoption as standard revisions in the definitions of the following terms: pick (addition of new Note); tenacity (addition of new Note 2); cloth; length, breaking; density; density, linear; atmosphere, standard; dew point; humidity; humidity, absolute; and humidity, relative.

Also recommended is a revision in Part B of Standard D 123 comprising a revised Table I on Man-Made or Synthetic Fibers and Monofilaments.

These revisions were prepared by Subcommittee B-2 and are appended hereto.[2]

Recommended Practice for A Universal System of Yarn Numbering (D 861 – 47):

The following revisions in this recommended practice are recommended for immediate adoption:

Revision of Note 1 and the deletion of Note 2 in line with the revised definitions for "density" and "density, linear" also being recommended at this time.

Revision of the value "4,960,536" to the rounded-off value "4,960,550" calculated from constants approved for engineering use by the American Standards Association and the British Standards Institution, and

To state all constants in the appendix to six significant figures in order to avoid possible errors in derived conversion factors.

All of these changes, although of a minor nature, have been incorporated in the revised recommended practice appended hereto.[3]

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Method of Test for Hard Scoured Wool in Wool in the Grease (D 584 – 47):

The committee recommends that the present tentative revision of Section 7 be adopted as standard and incorporated in this method.

Standard Specifications for Mechanical Roll Felt (D 944 – 49):

The committee recommends that the present tentative revision of Section 7 be adopted as standard and incorporated in these specifications.

Standard Methods of Testing Felt (D 461 – 49):

The committee recommends that the present tentative revision of the new Section 2 on Definition, the changes in Section 7 and the new Section S7 on Determination of pH of Felt be adopted as standard and incorporated in these methods.

WITHDRAWAL OF TENTATIVE

Committee D-13 recommends that the tentative listed below be withdrawn:

Tentative Specifications for Bleached Wide Cotton Sheeting (D 503 – 48 T).

EDITORIAL CHANGES IN TENTATIVES

Tentative Methods of Testing and Tolerances for Cotton Yarns (D 180 – 49 T):

Section 7 (d).—In the second line change the word "shall" to read "may."

The following revision to restore the requirement for the number of tests for appearance test, which had been previously omitted through error, is recommended by Committee D-13:

Section 11.—Add the following as a new Paragraph (*c*), with the present Notes 1 and 2 following thereafter:

> (*c*) *Number of Tests.*—Five tests shall be made on each case, bale, chain ball, or beam warp. If 80 per cent of the results meet the specified grade and the remaining 20 per cent do not fall below the next lower grade, the yarn shall be considered to be of the grade specified.

Tentative Methods of Testing and Tolerances for Continuous Filament Rayon and Estron Yarns (D 258 – 48 T), and

Tentative Methods of Testing and Tolerances for Rayon Tire Cord (D 885 – 46 T):

Editorial changes are necessary in these two standards in line with the changes recommended above in Standard D 861. In Section 13 (*b*) of D 258 and in Section 17 (*d*) of D 885 change the constant "4,464,483" to read "4,464,500" in the formula for calculating yards per pound of one denier yarn. In Section 13 (*b*) of D 258 and in Section 17 (*d*) of D 885 change the constant "4,960,537" to read "4,960,550" in the formula for calculating yards per pound of one grex yarn.

REPORT OF COMMITTEE D-14
ON
ADHESIVES[1]

Committee D-14 on Adhesives held two meetings during the year. A two-day meeting was held at A.S.T.M. Headquarters in Philadelphia on October 17 and 18, 1949 at which a talk on "The Evaluation of Test Procedures for Resin-Bonded Wood" was given by R. P. Hopkins. A spring meeting was held at A.S.T.M. Headquarters in Philadelphia on March 27 and 28, 1950, at which a talk was presented by N. V. Poletika on the "Testing of Adhesives for Wood" as presented in the educational program of the Timber Engineering Co.

At the present time, Committee D-14 consists of 78 members, of whom 32 are classed as producers, 31 as consumers, and 15 as general interest members.

At the March meeting the following officers were elected for the ensuing term of two years:

Chairman, F. W. Reinhart.
Vice-Chairman, G. Reinsmith.
Secretary, L. M. Perry.
Membership Secretary, John H. Wills.
Advisory Committee: J. F. Hamilton, G. Lipsey, F. J. Hanrahan.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-14 presented to the Society through the Administrative Committee on Standards proposed revisions of the Tentative Definitions of Terms Relating to Adhesives (D 907 - 47 T). This recommendation was accepted by the Standards Committee on September 22, 1949, and the revised tentative appears in the 1949 Book of A.S.T.M. Standards, Part 4.

On May 8, 1950, the Administrative Committee on Standards accepted the following recommendations submitted by Committee D-14:

Tentative Method of:

Test for Consistency of Adhesives (D 1084 - 50T).

Revision of Tentative Method of:

Test for Resistance of Adhesive Bonds to Chemical Reagents (D 896 - 46 T).

The new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 4.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentatives be continued in their present status without revision, pending further evaluation work and revision:

Tentative Methods of Test for:

Resistance of Adhesive Bonds to Chemical Reagents (D 896 - 46 T).[2]
Applied Weight Per Unit Area of Dried Adhesive Solids (D 898 - 47 T).[2]
Applied Weight Per Unit Area of Liquid Adhesive (D 899 - 47 T).[2]
Strength Properties of Metal to Metal Adhesives in Shear by Tension Loading (D 1002 - 49 T).[2]
Impact Strength of Adhesives (D 950 - 47 T).[2]

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[2] 1949 Book of A.S.T.M. Standards. Part 4.

Cleavage Strength of Metal to Metal Adhesive (D 1062 - 49 T).[2]

Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesive (D 904 - 46 T).[2]

It is also recommended that the Definitions of Terms Relating to Adhesives (D 907 - 49 T)[2] be retained as tentative.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (C. B. Hemming, chairman).—The Section on Tensile Strength is working on a simplified tensile method and is giving consideration to the determination of tensile strength of relatively low-strength adhesives. In addition to the three tentative methods for determining shear strength of wood and metal already published, the Section on Shear Strength has been conducting a round-robin for determining shear strength of plastic-to-plastic bonds. The Section also is considering the problem of determining shear strength of adhesives with extensible materials. The Section on Impact Strength is revising the Tentative Method of Test for Impact Strength of Adhesives (D 950 - 47T), and the revision is to go to letter ballot of Committee D-14. A round-robin on a method of test for Flexural-Shear Strength has been completed, and a tentative method is to be submitted to letter ballot of Committee D-14. The Section on Fatigue Strength has prepared two recommended practices for determining the fatigue properties of adhesives, which are to be submitted to letter ballot of Committee D-14. A Tentative Method of Test for Cleavage Strength of Metal to Metal Adhesives (D 1062 - 49 T) has been accepted by the Society. Round-robin work continues on peel testing. The Section on Creep and Flow is in process of being organized.

Subcommittee II on Analytical Tests (L. Repsher, chairman).—The subcommittee is currently working on methods of determining solids content of adhesives.

Subcommittee III on Permanence (R. F. Blomquist, chairman).—The Section on Effect of Moisture and Temperature has two test methods to be circulated for letter ballot of Committee D-14: Proposed Tentative Method of Test for Resistance of Adhesives for Wood to Cyclic Accelerated Service Conditions; and a Proposed Method for Continuous Exposure to Temperature and Humidity. The Section on Effect of Chemical Reagents prepared the revision of the Tentative Method of Test for Resistance of Adhesive Bonds to Chemical Reagents (D 896 - 46 T), recently approved by the Administrative Committee on Standards. The Section on Effect of Biological Factors on Permanence is to submit a Tentative Method for Testing Permanence of Adhesives in Various Biological Environments to letter ballot of Committee D-14. The Section on Effect of Light on Permanence is continuing round-robin work on the Tentative Recommended Practice for Determining the Effect of Artificial (Carbon-Arc Type) and Natural Light on the Permanence of Adhesive (D 904 - 46 T).

Subcommittee IV on Working Properties (D. L. Swayze, chairman).—The Section on Consistency prepared the Tentative Method of Test for Consistency of Adhesives: Method A: Cup Method recently approved by the Administrative Committee on Standards. Additional methods of determining consistency are also receiving consideration in this section. The Section on Tack and Blocking has worked out a blocking test which has passed letter ballot of the subcommittee, and the method is to be submitted to letter ballot of Committee D-14. Work on tack has progressed to the point where several instruments for measurement of tack are to be built and round-robin testing will proceed. A Recommended Practice for Determining Rate of Strength Development of Adhesives Bonds has passed letter ballot of Committee D-14, and the method is to be submitted to the Administrative Committee on Standards. An inquiry is being made to determine how widespread Tentative Methods D 898 and D 899 are being used, and how successful they have been. The Section on Storage and Working Life has a method of test ready for circulation in the subcommittee. Work continues on slippage and flow, penetration and gap filling properties of adhesives, but there is nothing significant to report.

Subcommittee V on Specifications (F. H. Bair, chairman).—Committee D-14 has set up a procedure to be followed for acceptance of specifications on special problems. The Section on General Utility Adhesives is in the stage of organizing, as are the sections on Packaging Adhesives, Label Adhesives, and Book Binding Adhesives. There is nothing to report on their work because the sections are relatively new. The Section on Adhesives for Acoustical Materials has circulated a specification within the section for comment, following which it will be circulated to the subcommittee for letter ballot.

Subcommittee VI on Nomenclature (F. Moser, chairman).—In addition to the set of definitions accepted by the Administrative Committee on Standards, work continues on other terms.

Subcommittee VII on Research (R. C. Platow, chairman) is conducting work on pure shear testing, fundamentals of adhesion and degradation. Under consideration also is a program on the problem of correlation between laboratory accelerated test cycles and normal outdoor weathering.

Subcommittee VII on Electrical Properties (H. A. Perry, chairman) is continuing work on insulation resistance and arc resistance. The subcommittee at the present time is trying to increase its membership.

This report has been submitted to letter ballot vote of the committee, which consists of 78 voting members; 66 members returned their ballots, of whom 63 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. W. REINHART,
Chairman.

G. W. KOEHN,
Secretary.

EDITORIAL NOTE

On January 19, 1951, the Administrative Committee on Standards accepted the recommendation of Committee D-14 that the Recommended Practice for Determining Strength Development of Adhesive Bonds be published as tentative. The new tentative recommended practice bears the designation D 1144 – 51 T and is available as a separate.

REPORT OF COMMITTEE D-15

ON

ENGINE ANTIFREEZES[1]

Committee D-15 on Engine Antifreezes held two meetings during the year: the first in New York, N. Y., on September 23, 1949, and the second in Washington, D. C., on February 17, 1950. The Advisory Committee and subcommittees have held meetings concurrently.

At its last meeting, the Advisory Committee activated Subcommittee V on Effect of Antifreeze on Rubber Hose and appointed T. E. Bieterman as chairman. This subcommittee plans to work jointly with Subsection B-III on Coolant System Hose, of the S.A.E.-A.S.T.M. Technical Committee on Automotive Rubber.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. R. Wolf.

Vice Chairman, D. B. Brooks.

Secretary, R. J. Harker.

Advisory Committee, Gus Kaufman, E. H. Keller, J. D. Klinger, J. C. Kratzer, L. W. Vollmer.

At present the committee is composed of 43 active members, of whom 21 are classified as producers, 10 as consumers, and 12 as general interest members.

NEW TENTATIVES

Committee D-15 recommends that one specification and five methods be accepted for publication as tentative, as appended hereto:[2]

Specifications for:

Hydrometer-Thermometer Field Tester for Engine Antifreezes,

Methods of Test for:

Reserve Alkalinity of Concentrated Engine Antifreezes,

Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer,

Water in Concentrated Engine Antifreezes by the Iodine Reagent Method,

Boiling Point of Engine Antifreezes, and

Ash Content of Concentrated Engine Antifreezes.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Freezing Point Determination (R. E. Mallonee, chairman) has made excellent progress in developing a test method for determining this important antifreeze property. It was decided at the Washington meeting, however, to await the outcome of some further collaborative work before submitting the test method as tentative. The information and test data obtained in this study have been assembled in the form of a technical paper by R. E. Mallonee and F. L. Howard, on "The Determination of Freezing Point of Engine Antifreezes."[4]

Subcommittee II on Antifreeze Field Testers (J. D. Klinger, chairman) prepared the new specifications for Hydrometer-Thermometer Field Tester for Engine Antifreezes and holds the honor of completing the first tentative to be approved by the committee and presented to the Society for publication.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The new specification and methods were accepted by the Society as tentative and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable: the results of the vote are on record at ASTM Headquarters.

[4] ASTM BULLETIN, No. 172, February, 1951, p. 43(TP57).

TABLE I.—RESULTS OF COOPERATIVE TESTS OF BOILING POINTS OF ENGINE ANTIFREEZE.

	Boiling Points, deg. Fahr.					
Sample No.	C-1-2		C-2-2		E-4-2	
Antifreeze, per cent	25	50	25	50	25	50
Aberdeen Proving Grounds	187.5	173.5	217.5	225.5	183.5	179.5
Carbide and Carbon Chemicals Corp.	187.0	173.5	217.0	225.5	185.0	178.5
Commercial Solvents Corp.	187.5	174.0	221.0	230.0	186.0	179.0
E. I. du Pont de Nemours and Co., Inc.	187.0	173.5	218.0	226.5	185.5	179.0
Gulf Research and Development Co.	187.5	173.5	218.5	228.0	185.0	178.0
National Bureau of Standards	187.0	174.0	216.5	225.0	185.0	178.0
Publicker Industries	187.0	174.0	217.0	227.0	184.5	178.5
Pure Oil Co.	186.0	172.0	216.5	225.5	184.0	178.0
Socony-Vacuum Oil Co.	188.0	174.0	218.5	227.5	184.5	179.0
Standard Oil Co. (Indiana)	186.0	172.5	217.5	226.0	184.5	178.0
The Texas Co.	185.5	172.5	217.0	224.5	184.5	177.5
U. S. Industrial Chemicals, Inc.	187.0	174.0	218.0	228.0	186.0	179.0
U. S. Naval Engineering Exper. Station	...	173.5	...	226.5	...	178.0
Mean Boiling Point, deg. Fahr.	186.9	173.4	217.8	226.6	184.8	178.5
Maximum Deviation from Mean, plus	1.1	0.6	3.2	3.4	1.2	1.0
Maximum Deviation from Mean, minus	1.4	1.4	1.3	2.1	1.3	1.0
Standard Deviation	0.73	0.67	1.24	1.51	0.75	0.595

NOTE.—All temperatures have been rounded off to the nearest 0.5 F.

The main committee has asked this subcommittee to investigate further the repeatability of the measurement of specific gravity of the antifreeze solutions after heating and cooling.

Subcommittee III on Physical Properties (R. E. Vogel, chairman) prepared the proposed Tentative Methods of Test for Boiling Point of Engine Antifreezes, and for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer. The boiling point method is applicable to both concentrated antifreeze and aqueous antifreeze solutions. Results of cooperative tests in 13 laboratories made in the development of this method are shown in Table I. The method for specific gravity is applicable only to concentrated antifreezes and is recommended as a tentative method at this time without precision limits. When established and approved by committee letter ballot, the precision limits will be presented for inclusion in the method. Collaborative work is planned to determine the suitability of this method for determining the specific gravity of aqueous antifreeze solutions.

TABLE II.—RESULTS OF COLLABORATIVE WORK ON DETERMINATION BY THE KARL FISCHER METHOD OF WATER CONTENT OF ENGINE ANTIFREEZE.

Laboratory	Water Content by Direct Titration in Methanol, per cent by weight				
	Sample No.				
	B-2-1	C-2-2	D-3-2	F-1-1	C-1-2
Commercial Solvents Corp.	5.4	4.1	7.4	0.4	0.2
E. I. du Pont de Nemours and Co., Inc.	5.6	4.1	7.0	0.4	...
Linde Air Products Co.	5.5	4.1	7.5	0.5	0.3
Shell Oil Co.	4.6	4.0	7.1	0.4	0.3
Standard Oil Co. (Indiana)	5.4	4.1	7.2	0.4	...
The Texas Co.	5.8	4.4	5.8	0.4	...
Average	5.4	4.1	7.0	0.4	0.3
High	5.8	4.4	7.5	0.5	0.3
Low	4.6	4.0	5.8	0.4	0.2
Range	1.2	0.4	1.7	0.1	0.1

Subcommittee IV on Chemical Properties (R. J. Harker, chairman) has completed collaborative test work on three methods which have been approved by letter ballot of the committee and are recommended for publication as proposed tentative methods. These methods are applicable to undiluted antifreezes only.

The new Method of Test for Water Content of Concentrated Engine Antifreezes is based upon the Karl Fischer method. The data obtained by the six cooperating laboratories as shown in Tables II and III, indicated that very satisfactory reproducibility may be obtained through the use of this test procedure.

TABLE III.—COMPARISON OF RESULTS BETWEEN TITRATION IN METHANOL AND PYRIDINE IN WATER CONTENT DETERMINATION.

Laboratory	Water Content, per cent by weight					
	Sample No. B-2-1		Sample No. D-3-2		Sample No. F-1-1	
	Methanol	Pyridine	Methanol	Pyridine	Methanol	Pyridine
E. I. Du Pont de Nemours and Co., Inc.	5.6	5.7	7.0	7.1	0.8	0.8
Linde Air Products Co.	5.7	5.4	7.5	7.0	0.5	0.5
Shell Oil Co.	5.3	5.3	7.4	7.4	0.5	0.4

NOTE.—Titration in pyridine instead of methanol minimizes side reactions when carbonyl compounds are present in the sample.

The collaborative test study of the new method for the Determination of Ash Content of Concentrated Engine Antifreezes indicated that there was no advantage in precision and reproducibility to be obtained from determining sulfated ash. The results indicated that the simpler ashing procedure proposed is adequate for antifreeze materials.

The subcommittee has also prepared a new method for the Determination of the Reserve Alkalinity of Concentrated Engine Antifreezes, following the completion of a series of cooperative tests. It was decided that the titration shall be a pH value of 5.5.

A new method for the Determination of the pH of Engine Antifreezes has been developed as a modification of the present Tentative Method for Determination of the pH of Aqueous Solutions with the Glass Electrode (E 70 – 46 T). When completed this method

TABLE IV.—RESULTS OF COOPERATIVE STUDY OF ASH TEST METHOD.

Laboratory	Sample B-2-1		Sample C-1-2		Sample C-2-2		Sample D-3-2		Sample F-1	
	Direct Ash	Sulfated Ash	Direct Ash	Sulfated Ash	Direct Ash	Sulfated Ash	Direct Ash	Sulfated Ash	Direct Ash	Sulfated Ash
E. I. du Pont de Nemours and Co.	0.76 0.80	0.98 0.98	0.34 0.37	0.64 0.63	0.70 0.71	1.12 1.08	0.10 0.10	0.15 0.15	0.13 0.12	0.14 0.15
The Texas Co.	0.62 0.64	1.00 0.98	0.27 0.28	0.54 0.54	0.64 0.66	0.98 0.94 0.93	0.07 0.07	0.16 0.15	0.10	0.13
Commercial Solvents Corp.	0.72 0.79	0.89 0.89	0.30 0.33	0.65 0.64	0.71 0.75	1.03 1.04	0.08 0.08	0.17 0.15	0.15 0.15	0.12 0.14
Packard Motor Car Co.	0.71	0.99	0.28	0.55	0.67	0.96	0.04[a]	0.11	0.12	0.13
Gulf Research and Development Co.	0.72 0.72	0.97 0.91	0.37 0.33	0.62 0.60	0.71 0.71	1.15 1.19	0.10 0.07	0.15 0.15	0.14 0.14	0.14 0.13
Linde Air Products Co.	0.67 0.65	1.00 0.95	0.25 0.25	0.53 0.51	0.68 0.71	1.05 0.97	0.10 0.08	0.12 0.13	0.13 0.12	0.13 0.13
Publicker Industries	0.85[a]	0.88	0.36	0.58	0.81[a]	0.68[a]	...	...	...	...
Grand Average	0.70	0.95	0.31	0.59	0.69	1.03	0.085	0.14	0.13	0.13
Maximum Deviation from Mean: plus	0.10	0.05	0.06	0.06	0.06	0.16	0.02	0.03	0.03	0.03
Maximum Deviation from Mean: minus	0.03	0.07	0.06	0.08	0.06	0.10	0.01	0.03	0.03	0.01

[a] Results discarded.

TABLE V.—RESULTS OF COOPERATIVE STUDY OF RESERVE ALKALINITY TEST METHOD.

	Reserve Alkalinity, ml. of 0.1 *N* HCl required to titrate 10-ml. sample of concentrated antifreeze to pH value indicated.																			
Sample No.	B-2-1				C-1-2				C-2-2				D-3-2				F-1-1			
pH value	6.0	5.5	5.0	4.5	6.0	5.5	5.0	4.5	6.0	5.5	5.0	4.5	6.0	5.5	5.0	4.5	6.0	5.5	5.0	4.5
Packard Motor Car Co.	12.52	12.65	12.72	12.85	1.98	2.03	2.15	2.50	15.80	15.93	16.00	16.10	0.22	0.25	0.29	0.40	1.15	1.23	1.27	1.33
Linde Air Products Co.	12.92	12.99	13.02	13.08	2.14	2.22	2.80	4.18[a]	16.20	16.30	16.36	16.41	0.25	0.31	0.44	0.65	0.96	1.06	1.18	1.31
U. S. Industrial Chemicals	12.60	12.72	12.76	12.82	2.06	2.10	2.15	2.50	15.74	15.86	15.92	15.97	0.17	0.20	0.25	0.33	0.85	1.05	1.12	1.17
E. I. du Pont de Nemours & Co., Inc.	13.10	13.15	13.20	13.25	2.05	2.10	2.20	2.45	16.30	16.35	16.40	16.50	0.30	0.40	0.45	0.95[a]	1.05	1.20	...	1.25
The Texas Co.	12.90	13.00	13.00	13.00	2.10	2.20	2.30	2.80	15.80	16.00	16.10	16.30	0.20	0.20	0.30	0.40	1.20	1.30	1.40	1.40
Chrysler Corp.	12.54	12.99	13.02	13.06	2.09	2.18	2.29	2.85	16.37	16.49	16.58	16.71	0.23	0.29	0.36	0.51	1.05	1.16	1.21	1.27
Gulf Research and Development Co.	12.71	12.77	12.82	12.92	2.22	2.26	2.46	3.52	15.87	16.00	16.07	16.11	0.26	0.30	0.33	0.46	1.13	1.21	1.23	1.27
Publicker Industries	11.00[a]	11.80[a]	12.50	13.00	2.40	2.60	3.00	4.20[a]	15.60	16.10	18.10[a]	23.20[a]	...	...	...	...	3.10[a]	3.40[a]	3.80[a]	5.10[a]
Commercial Solvents Corp.	12.53	12.64	12.73	12.83	2.22	2.31	2.43	2.69	16.03	16.13	16.15	16.27	0.47[a]	0.56[a]	0.70[a]	0.86[a]	0.94	1.06	1.14	1.19
Mean Reserve Alkalinity	12.72	12.86	12.86	12.98	2.14	2.22	2.42	2.62	15.97	16.13	16.20	16.30	0.23	0.28	0.35	0.46	1.04	1.16	1.22	1.27
Maximum Deviation																				
from Mean plus	0.38	0.29	0.29	0.27	0.26	0.38	0.53	0.90	0.33	0.36	0.38	0.41	0.03	0.12	0.10	0.19	0.16	0.14	0.18	0.13
minus	0.20	0.22	0.36	0.16	0.16	0.19	0.27	0.15	0.37	0.27	0.28	0.30	0.05	0.08	0.10	0.13	0.19	0.09	0.10	0.10

[a] Results discarded.

will be submitted to letter ballot of the committee for publication as tentative.

Subcommittee VI on Simulated and Actual Service Testing (E. H. Keller, chairman) has prepared a more detailed outline of its work, which is appended to this report.

Subcommittee VII on Specifications (J. M. Clark, chairman) met on September 22, 1949, to review its work. This subcommittee does not currently have available sufficient data to prepare specifications for engine antifreezes. However, it has again recommended to the main committee that the projects under investigation by the other subcommittees be pursued actively to provide necessary information so that specifications can be prepared at an early date.

Study Group on Sampling (R. E. Mallonee, chairman) is engaged in the preparation of standard methods for sampling and preparing engine antifreezes for test. This information will be of considerable value to the subcommittees in developing test methods that reflect antifreeze properties under actual operating conditions.

This report has been submitted to letter ballot of the Committee, which consists of 43 voting members; 35 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. R. WOLF,
Chairman.

E. H. KELLER,
Secretary.

REPORT OF SUBCOMMITTEE VI ON SIMULATED AND ACTUAL SERVICE TESTING

Subcommittee VI on Simulated and Actual Service Testing is conducting cooperative test programs on two methods of test for the corrosion and foaming tendencies of engine antifreezes. In addition, it is preparing a short article which deals with certain phases of antifreezes useage, not covered in other publications on the subject.

The subcommittee is well advanced in its cooperative evaluation of "glassware type" corrosion tests for antifreeze solutions. It was agreed that each of the ten cooperating laboratories would use its own test method to evaluate nine aqueous antifreeze materials and water. No restrictions were placed upon test methods or conditions of operation. Antifreezes are being rated by each laboratory in the order of their effectiveness in reducing the corrosion of cooling system metals.

The committee has agreed that the corrosion and foaming properties of antifreeze solutions can be evaluated with accuracy only in an automobile cooling system. It is well aware, however, of the disadvantages inherent in such a test procedure, involving the use of actual vehicles. The number of units required, the expense of the equipment, and cost of trained personnel to conduct the test would undoubtedly limit its use to large, well-equipped testing laboratories.

In order to simplify the procedure and reduce the cost of equipment to a minimum, the committee is conducting a series of cooperative tests in a circulating apparatus, containing some automotive cooling system parts. The apparatus and procedure used are a modification of the Simulated Service Test Unit developed by the National Bureau of Standards. A description of the apparatus submitted by the Bureau to this committee in January, 1948, follows:

> The apparatus used in the simulated service corrosion tests, which this Bureau is conducting for Government agencies, consists of a circulating system, including a motor-driven automobile coolant pump connected by radiator hose and pipe to a 5-gal. steel tank and a commercial automotive radiator. The pump is designed to run at different speeds, corresponding to certain road speeds of automobiles. The steel tank rests on an electric hot plate, the temperature of which can be controlled. Through a cap in the top of a standpipe screwed onto the 5-gal. tank, weighed specimens of different types of metals commonly used in automotive engine cooling systems are suspended in the antifreeze coolant. Successive weighings of each metal specimen will show whether or not the antifreeze coolant is deleterious to the different metal specimens. Radiator and ambient temperatures are regulated to give a normal automotive engine operating temperature, permissible for the type of antifreeze under test, and generally between 170 and 180 F.
>
> The metal test specimens are 1⅛ in. squares, approximately 0.030-in. in thickness, of aluminum, brass, copper, brass soldered to copper, steel, and cast iron, each with a 3/16-in. hole drilled in the center. The specimens are polished, thoroughly cleaned, and each weighed on a chemical balance. Initial weights are recorded and the specimens are strung on a threaded brass eyebolt, with a ⅜-in. diameter tpacer tube ½ in. long between specimens. The whole assembly was locked securely together, thus assuring good electrical contact. The eyebolt assembly was attached to one end of a brass rod and the other end secured to a removable cap plate on

the standpipe, allowing the metal specimens to be completely immersed in the 5-gal. tank when the circulating system was filled to the proper level with the test solution. The strength of the antifreeze test solution used is such as to have a freezing point of 0 F.

The minimum time a cooling system test should run is dependent upon the corrosion action of the antifreeze solution upon the different metal specimens in the circulating system.

The subcommittee hopes to be able to present a laboratory test procedure that will consistently differentiate between safe and deleterious antifreeze products, although some correlation with actual operating experience will undoubtedly be necessary.

Respectfully submitted on behalf of the subcommittee,

E. H. KELLER,
Chairman.

REPORT OF COMMITTEE D-16
ON
INDUSTRIAL AROMATIC HYDROCARBONS[1]

The initial objective of Committee D-16 on Industrial Aromatic Hydrocarbons, the establishment of specifications and methods of test for the major industrial aromatic hydrocarbons as standards of the Society, has been accomplished, and the published standards are enjoying increasingly wide circulation. Recent activity of the committee has been concerned principally with the second phase of the work, which consists of the correlation of test methods and apparatus with similar standards sponsored by other committees with the object of minimizing duplication and keeping methods and laboratory apparatus to a minimum. To this end the committee is recommending in this report certain revisions in the standards under its jurisdiction.

Since there are related chemicals other than hydrocarbons that are of industrial importance and which are not now covered by A.S.T.M. standards, the committee has indicated the need for an extension of its scope and has appointed a special subcommittee to review the matter.

Consideration is being given to available A.S.T.M. methods of test for sulfur and copper corrosion which were prepared by or are under consideration by other committees of the Society, again with the object of minimizing duplication of effort.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in 12 standard specifications and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society. These changes in the specifications, as well as those in Method D 850, are in line with current endeavors to coordinate A.S.T.M. distillation tests, looking toward the establishment of a universal A.S.T.M. distillation method.

The revision consists of adding to each of the following specifications a paragraph under the section on methods of testing requiring the material to be sampled in accordance with the Tentative Methods of Sampling Petroleum and Petroleum Products (A.S.T.M. Designation: D 270).[2] In addition a reference is to be added to each specification to the A.S.T.M. thermometer to be used in the distillation test as indicated below:

Standard Specifications for:[3]

Nitration Grade Benzene (D 835 – 47), including a reference to A.S.T.M. Solvents Distillation Thermometer 40 C.,

Industrial Grade Benzene (D 836 – 47), including a reference to thermometer 40 C.,

Industrial 90 Benzene (D 837 – 47), including a reference to thermometer 40 C.,

Refined Solvent Naphtha (D 838 – 47), including a reference to thermometer 42 C.,

Crude Light Solvent Naphtha (D 839 – 47), including a reference to thermometer 42 C.,

Crude Heavy Solvent Naphtha (D 840 – 47), including a reference to thermometer 42 C.,

Nitration Grade Toluene (D 841 – 47), including a reference to thermometer 41 C.,

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] 1950 Supplement to Book of ASTM Standards, Part 5.

[3] 1949 Book of A.S.T.M. Standards, Part 5.

Industrial Grade Toluene (D 842 – 47), including a reference to thermometer 41 C.,
Nitration Grade Xylene (D 843 – 47), including a reference to thermometer 41 C.,
Industrial Grade Xylene (D 844 – 47), including a reference to thermometer 42 C.,
Five-Degree Xylene (D 845 – 47), including a reference to thermometer 41 C.,
Ten-Degree Xylene (D 846 – 47), including a reference to thermometer 41 C.

The committee also recommends the following revision for immediate adoption in the Standard Method of Test for Distillation of Industrial Aromatic Hydrocarbons (D 850 – 47):[3]

Section 2 (b).—Change the thermometer requirements to read as follows:

(*b*) *Thermometer*.—(*1*) The A.S.T.M. Solvents Distillation Thermometer used in the test shall be as prescribed in the specifications for the material being tested. The thermometer shall conform to the requirements prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).[3] Table I shows the ranges of the several available A.S.T.M. solvents distillation thermometers suitable for testing industrial aromatic hydrocarbons.

TABLE I.—A.S.T.M. THERMOMETERS FOR DISTILLATION TEST OF INDUSTRIAL AROMATIC HYDROCARBONS.

A.S.T.M. Designation	Name	Range deg. Cent.	Subdivision, deg. Cent.
39C......	solvents distillation	48 to 102	0.2
40C......	solvents distillation	72 to 126	0.2
41C......	solvents distillation	98 to 152	0.2
42C......	solvents distillation	95 to 255	0.5

(*2*) As an alternate, thermometers of the following description may be used: Thermometers of the mercury, etched glass stem type, graduated in 0.2 C. intervals, and having a range of 70 to 120 C. for benzene and toluene and a range of 110 to 160 C. for xylene. Graduations shall be numbered at each multiple of 2 C. The thermometers shall be made of a suitable quality of glass so as not to change readings under conditions of use. They shall be provided with an expansion chamber filled with nitrogen and with a ring at the top. The thermometers shall conform to the following dimensions:

Total length	**not more than 305 mm.**
Length of bulb	**not more than 20 mm.**
Distance from bottom of bulb to graduation line at:	
70 C., for thermometer range of 70 to 120 C	**80 to 100 mm.**
110 C., for thermometer range of 110 to 160 C	**80 to 100 mm.**
Graduations per inch	**not more than 35**
Diameter of stem	**5 to 7 mm.**
Diameter of bulb (not more than diameter of stem)	**5 to 7 mm.**

The scale error at any point of the scale shall not exceed 0.2 C. when the thermometer is compared at total immersion with a similar thermometer calibrated at total immersion by the National Bureau of Standards and proper corrections are applied. It is preferable that thermometers do not have a contraction chamber situated between the bulb and the lowest graduation. However, if such a bulb is present, its lowest point shall be not more than 1 mm. above the top of the main reservoir.

Section 2 (e).—Change the condenser requirements to read as follows:

(*e*) *Condenser*.—Either of the following two condensers may be used:

(*1*) The condenser specified in the Standard Method of Test for Distillation of Gasoline Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),[3] or

(*2*) As an alternative, the condenser tube may consist of a straight glass tube 600 to 610 mm. in length and 12 mm. in inside diameter, of standard wall thickness (about 1.25 mm.) with the exit end cut off square and ground flat. It shall be set in a cooling trough so that at least 15 in. of the tube is in contact with the water. Clearance between the condenser tube and any parallel side of the trough shall be not less than $\frac{3}{4}$ in. The water in the cooling trough shall be maintained at 10 to 20 C. (50 to 70 F.). This may be done by adding ice to the water or by circulating chilled water through the trough. The trough shall be so mounted that the condenser tube is set at an angle of 75 deg. with the vertical.

Section 6 (b).—Add the following: "If the alternate thermometers (total immersion) of Section 2 (b) are used,........"

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the Tentative Method of Test for Thiophene in Benzene (D 931 – 47 T)[3] be approved for reference to letter ballot of the Society for adoption as standard.

EDITORIAL CHANGE

The following editorial change is recommended in the Standard Method of Test for Solidifying Point of Benzene (D 852 – 47):[3]

Section 2 (*e*).—Change to read as follows:

> (*e*) *Thermometer.*—An A.S.T.M. Benzene Freezing Point Thermometer having a range of −0.6 to +10.4 C. and conforming to the requirements for thermometer 53C as prescribed in A.S.T.M. Specifications E 1.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 38 voting members; 23 members returned their ballots, of whom 21 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

D. F. GOULD,
Chairman.

R. B. GREENE,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-17

ON

NAVAL STORES[1]

Committee D-17 on Naval Stores held one meeting during the year: on June 29, 1949, at Atlantic City, N. J., in connection with the Annual Meeting of the Society. At the time of the 1948 meeting it was tentatively decided to dispense with separate subcommittee meetings in view of the general interest of all committee members in the overall program of work of the various subcommittees. In accordance therewith, the work of each subcommittee was subjected to a full discussion in the committee meeting.

The summary of known methods for determining softening point of resins, which had been incorporated in the Appendix to the 1948 Report of Committee D-17,[2] was made available in separate pamphlet form at the 1949 Meeting.

A nominating committee, consisting of W. D. Pohle, G. R. Tennent, and W. C. Smith, was appointed by the chairman. The following officers and members of the Advisory Committee were elected at the 1950 Annual Meeting:

Chairman, V. E. Grotlisch.

Vice Chairman, J. L. Boyer.

Secretary, W. A. Kirklin.

Advisory Committee, J. P. Bain, R. P. Chapman, R. E. Price, I. E. Knapp, G. R. Tennent, and Authur Pollak.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-17 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Methods of Testing Tall Oil (D 803 - 44 T) be revised. This recommendation was accepted by the Standards Committee on September 22, 1949, and the revised tentative methods appear in the 1949 Book of A.S.T.M. Standards, Part 4.

NEW TENTATIVE

The committee recommends for acceptance and publication as tentative the Methods of Test for Rosin Oils, as they appear in Appendix II of the 1949 Report,[3] with the following minor changes:

Section 5 (*b*).—In item (*1*), insert 100 F. as an alternate test temperature.

Section 8.—Substitute "302" in place of "346" for molecular weight of rosin acids. In Eq. 1 substitute "0.302" for "0.346." Change the factor "0.617" to "0.539" in Eq. 2 and in the Note.

REVISION OF TENTATIVE

Tentative Method of Test for Water in Liquid Naval Stores (**D 890 - 46 T**).[4]—On the basis of experience with this method by several members, the committee recommends revision of the method so as to provide for preparation of the Karl Fischer reagent (iodine-pyridine-sulfur dioxide) in two stable solutions that do not become water-reactive until they are brought together at the time of making the test. This involves a major revision of the method, and the revised method is appended to this report.[5]

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 457 (1948).

[3] *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 469 (1949).

[4] 1949 Book of A.S.T.M. Standards, Part 4.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the following tentative be approved for reference to letter ballot of the Society for adoption as standard, with revisions as indicated:

Tentative Method of Test For Volatile Oil in Rosin (**D 889 - 46 T**).[4]—This method appears satisfactory, with but two minor changes, as follows:

Section 3 (a).—In the first sentence, substitute the words "insert an ebullition tube" in place of "a few glass beads."

Section 3 (b).—Delete the last sentence of this paragraph.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman).—A member of this subcommittee, serving as chairman of a task group on Softening Point, of Committee E-1, Subcommittee 9, has reported progress in the efforts to combine the two A.S.T.M. Ring-and-Ball Softening Point Methods, D 36 and E 28, into one method acceptable to all the interested technical committees. Editorial changes in two sections of Method E 28 have been suggested by a member of the subcommittee to eliminate possibility of misinterpretation of directions for preparing a sample of rosin for the test and the first steps in conducting the test.

Subcommittee III on Volatile Oil in Rosin (S. R. Snider, chairman).—On the basis of further work on the tentative method for determining volatile oil in rosin, recommendation has been made that the method be adopted as standard.

Subcommittee IV on Chemical Analysis of Rosin (W. D. Pohle, chairman).—Collaborative work has been done on the tentative methods for acid number and saponification number of rosin. The potentiometric methods for titration in these methods, developed for use with dark rosins, have proven entirely reliable and the tentatives have been recommended for adoption as standard.

Work on the Tentative Method of Test for Unsaponifiable Matter in Rosin (D 1065 - 49 T) is to be continued. A member of the subcommittee has developed a method using standard separatory funnels for extraction of unsaponifiables from the solution, which is offered for consideration as an alternate procedure to the present tentative method. This proposed substitute method is appended[7] to this report as information only.

Subcommittee V on Tall Oil (A. Pollak, chairman).—Through the efforts of the chairman and several members of the subcommittee, it was possible to complete the revisions and modifications in the Tentative Methods for Testing Tall Oil (D 803 - 49 T) for inclusion in the 1949 Book of A.S.T.M. Standards. Plans are now being developed for preparation of synthetic tall oil mixtures of known fatty and rosin acid content, for further collaborative work on the methods.

Subcommittee VII on Terpene Hydrocarbons and Pine Oil (R. E. Price, chairman).—Members of the subcommittee have studied the Tentative Method of Test for Water in Liquid Naval Stores (D 890 - 46 T) and this has resulted in the proposed revision of this tentative method, as appended to this report.[5]

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[5] The revised method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 4.

[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[7] See p. 391.

Subcommittee IX on Definitions (J. E. Boyer, chairman).—This subcommittee calls attention to the manufacture of a new kind of rosin made from tall oil by fractional vacuum distillation, by which the fatty acids are separated from the rosin acids. The definition of rosin as given in Standard Definitions D 804 will need revision to include the new kind of rosin. The U. S. Department of Agriculture is now considering a new designation or standard of identity for this rosin from tall oil, on the basis of information developed at a hearing held on May 9, 1950. Points of similarity and dissimilarity in chemical composition, as compared with normal gum and wood rosins, were discussed. Pending an announcement by the Secretary of Agriculture, the manufacturer has been authorized to describe the new rosin as "sulfate wood rosin."

This report has been submitted to letter ballot of the committee, which consists of 40 voting members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

APPENDIX

PROPOSED ALTERNATE METHOD OF TEST FOR UNSAPONIFIABLE MATTER IN ROSIN[1]

This is a proposed method and is published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. This method of test is intended for use as an alternate procedure for determining the percentage of unsaponifiable matter in rosin, when the special extraction apparatus described in Section 2 of the Tentative Method of Test For Unsaponifiable Matter in Rosin (A.S.T.M. Designation: D 1065)[2] is not available, or when duplicate tests must be made in shorter time than is required for the continuous extraction method.

Reagents

2. For descriptions of the reagents required, reference should be made to Method D 1065.

Procedure

3. (*a*) Weigh 4.00 ± 0.001 g. of the freshly crushed sample into a 250 to 300-ml. Erlenmeyer flask (alkali-resistant glass preferred) having a standard-taper neck. Add 50 ml. of KOH solution (33 g. per l.), connect to a water-jacketed condenser, and reflux gently for 30 min. Disconnect the condenser and evaporate gently to about 15 ml. to expel alcohol. Add 10 ml. of water and again boil gently until the odor of alcohol is not detected at the mouth of the flask.

(*b*) Transfer the contents of the flask quantitatively to a 300-ml. separatory funnel *A*, rinsing with three 20-ml. portions of hot water. Cool the flask under tap water and add 50 ml. of ether. Shake well, and allow the aqueous and ether layers to separate. Draw off the aqueous layer into a second separatory funnel *B*. Then add 5 ml. of water to funnel *A* without shaking, again drawing the aqueous layer into funnel *B*.

(*c*) Extract the solution in funnel *B* with 50 ml. of fresh ether, with thorough shaking, and, after separation, draw off the aqueous layer into a third separatory funnel *C*. Add 5 ml. of water to funnel *B*, without shaking, and again draw the aqueous layer into funnel *C*.

(*d*) Run the ether extract in funnel *B* into funnel *A*, washing the stem of funnel *B* with 5 ml. of ether into funnel *A*. Close the stopcock and wash interior of funnel *B* into funnel *A* with 5 ml. of ether.

(*e*) Extract the aqueous solution in funnel *C* a third time with 50 ml. of ether, as before. Draw off and discard the aqueous portion and combine the ether with that in funnel *A*. Add 50 ml. of 1 per cent aqueous alkali solution, shake thoroughly, allow to separate, and discard the aqueous layer. Then wash the ether layer twice with 50-ml. portions of water.

(*f*) Draw the combined ether extracts into a 300-ml. beaker, in which a layer of about 10 g. of anhydrous powdered Na_2CO_3 has been spread over the bottom. Wash the funnel with 10 ml. of ether, adding the ether to the beaker. Stir

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[1] This proposed method is under the jurisdiction of the A.S.T.M. Committee D-17 on Naval Stores. Published as information, June, 1950.

[2] 1949 Book of A.S.T.M. Standards, Part 4.

(APPENDIX)

the ether and Na_2CO_3 thoroughly and allow to stand for 10 to 15 min., with occasional stirring. After final separation, carefully transfer the ether to a weighed beaker of similar size. Break up any caked Na_2CO_3 in the first beaker with a stirring rod, wash with two 15-ml. portions of ether, and add the ether to the weighed beaker.

(*g*) Place the beaker on a *warm* steam bath and evaporate gently until the ether is expelled, taking care to prevent condensation of water on the inside of the beaker. When ether odor is completely removed from the beaker, place it in a desiccator to cool, and weigh. The weight of contents of the beaker is the "net washed extract."

(*h*) Dissolve the extract in 50 ml. of neutral alcohol, with the addition of a small quantity of benzene, if necessary, to effect complete solution. Add 1 ml. of phenolphthalein indicator, and titrate any free acid formed by hydrolysis with 0.1 *N* alcoholic KOH solution.

Calculation and Report

4. Calculate the unsaponifiable matter in the rosin as directed in Section 6 of Method D 1065.

REPORT OF COMMITTEE D-18

ON

SOILS FOR ENGINEERING PURPOSES[1]

Committee D-18 on Soils for Engineering Purposes held two meetings during the year. The first meeting was held during the Annual Meeting at Atlantic City, N. J., on June 29, 1949. Thirty-three members and eleven visitors were present. At that time a technical session on soils was held at which time the following papers[2] were presented:

> "The Procedure and Significance of Chemical and Mineralogical Tests in Soils" by T. W. Lambe and Dr. V. F. B. deMello,
>
> "Subsurface Exploration by Geophysical Methods" by E. R. Shepard, and
>
> "Study on Mechanical Oscillators" by R. K. Bernard.

One additional paper[3] was presented at the close of the main meeting. This paper was entitled:

> "Thixotropic Strength Regain of Clays" by Louis Berger and J. P. Gnaedinger.

The committee met at San Francisco, Calif., on October 11, 1949, during the Pacific Area National Meeting. There were twelve members and five visitors present. A technical session on soils was held and the following papers were presented:

> "Effect of Rock Content and Placement Density on Consolidation and Related Pore Pressure in Embankment Construction" by Harold J. Gibbs,[4]
>
> "Electrical Resistivity Method Applied to the Investigation of Construction Materials Deposits" by E. A. Abdun-Nur and D. Wantland,[5] and
>
> "Studies of Construction Problems and Methods of Compacting Different Type Soils to Optimum Density" by A. W. Root, W. S. Maxwell, and T. E. Stanton.[6]

The Executive Subcommittee also held a meeting in Washington, D. C., on December 12, 1949. Six members attended.

During the June, 1949, meeting, the chairman regretfully reported to the committee the death of C. A. Hogentogler, past chairman of Committee D-18. A letter which expressed the deepest sympathies of the committee members was sent to Mrs. Hogentogler. This letter was placed in the minutes of the June 29, 1949, committee meeting.

During the year, 35 new members, representing individuals or organizations were admitted to the committee. The total committee membership is now 139, which includes 92 voting memberships and 17 consulting memberships.

The Special Subcommittee on Procedures for Testing Soils, which was organized to revise the 1944 edition of the compilation *Procedures for Testing Soils*, completed its work in revising old procedure items and in securing additional procedure items for this publication. Fifty-two new or revised items were secured from the membership. The revised compilation has been edited and is being prepared for printing. Copies are expected to be available for distribution this summer.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 49 (1949).

[3] ASTM Bulletin, No. 160, September, 1949, p. 64 (TP 162).

[4] See p. 1343.

[5] See p. 1364

[6] Published in the *Western Construction News*, May 15, 1950.

TENTATIVES CONTINUED WITHOUT REVISION

Committee D-18 recommends that the following tentatives be continued in their present status without revision:

Definition of Terms and Symbols Relating to Soil Mechanics (**D 653 - 42 T**),

Method of Test for Moisture-Density Relations of Soils (**D 698 - 42 T**),

Method of Testing Soil-Bituminous Mixtures (**D 915 - 47 T**), and

Method of Test for Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (**D 916 - 47 T**).

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee D-18 recommends for immediate adoption the following revisions and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society:

Standard Methods of Surveying and Sampling Soils for Highway Subgrades (**D 420 - 45**).[7]—

Section 1.—Change the beginning of the first sentence to read as follows:

1. The purpose of the subgrade survey is to furnish the Engineer with significant information for design or decision on the following subjects:

Section 2.—Add the following as Paragraph (*a*):

2. (*a*) The subgrade survey should determine what materials are involved and where the materials are located. More specifically, the information should include location and depths of all soil types, complete description and classification of the different soils and general conditions of density and moisture of the different soils encountered. The search for the information consists of related but separate field operations enumerated as follows:

(*1*) Field reconnaissance.

(*2*) Subsurface exploration test holes.

(*3*) Description, classification, and logging of test holes.

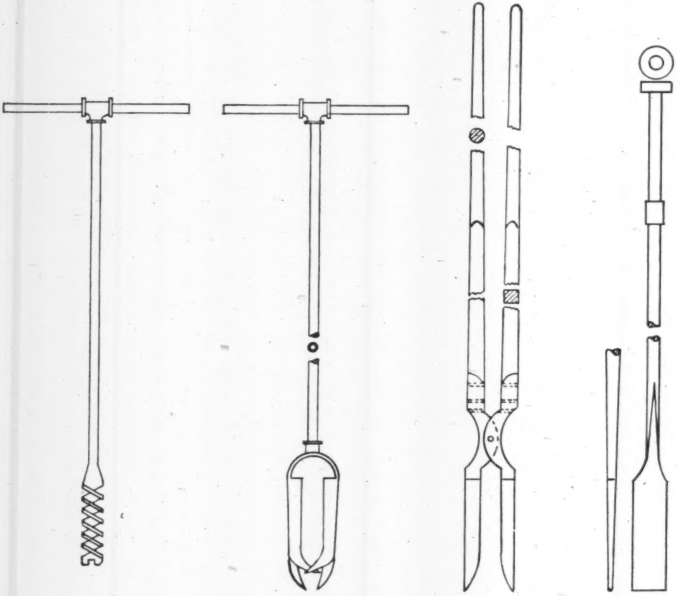

FIG. 1.—Equipment for Obtaining Samples of Soil.

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[7] 1949 Book of A.S.T.M. Standards, Part 3.

(*4*) Sampling for inspection and testing.
(*5*) Summarizing data into soil profile (which provides a graphic record of survey).

Change the present Paragraph 2 to 2(*b*).

Section 3.—Change to read as follows:

3. The following equipment is required to make a subgrade survey:

(*a*) Drilling equipment suitable for making test borings. Some of the hand drills and augers satisfactory for this purpose are illustrated in Fig. 1; also, power augers, if available, may be used for making test borings.
(*b*) Two small pipe wrenches.
(*c*) Pick.
(*d*) Shovel.
(*e*) A supply of sample containers including tightly woven cloth bags and pint size jars or cans.
(*f*) A supply of tags and labels for identifying samples.
(*g*) A ball of twine.
(*h*) One engineer's level.
(*i*) One hand level.
(*j*) One level rod.
(*k*) One 100-foot metallic tape.
(*l*) One 6-foot folding rule.
(*m*) One roll of 20 in. cross section paper, 10 divisions to the inch each way.
(*n*) Field note books.
(*o*) A supply of survey stakes.
(*p*) One camera and supply of films.
(*q*) A supply of keel.
(*r*) Canvas or other suitable material to be used in quartering samples.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

SUBCOMMITTEE ACTIVITIES

Subcommittee G-3 on Nomenclature and Definitions (C. R. Foster, chairman) has recommended that the tentative standard D 653 – 42 T, Definition of Terms and Symbols Relating to Soil Mechanics be continued in its present status until the committee has completed the revisions on which they are now working. C. R. Foster replaced E. A. Abdun-Nur as chairman of the subcommittee and W. K. Boyd resigned from the subcommittee and was replaced by Mr. Abdun-Nur.

Special Subcommittee on Procedures for Testing Soils (M. D. Catton, chairman). —This subcommittee completed its work on the revised edition of "Procedures for Testing Soils" and was disbanded by action of the Executive Committee in December, 1949.

Subcommittee R-1, Surface and Subsurface Reconnaissance (L. E. Gregg, chairman) held two meetings during the year and completed its organization. Two papers were sponsored on technical programs, one for the Annual Meeting and one for the Pacific Coast Meeting. This subcommittee is planning a symposium dealing with all phases of surface and subsurface reconnaissance for the 1951 Annual Meeting.

Subcommittee R-2 on Borings and Sampling for Soil Investigation (J. O. Osterberg, chairman) met during the Pacific Coast Meeting. C. W. Allen resigned as chairman of this subcommittee and was replaced by J. O. Osterberg. The revision of Standard Methods of Surveying and Sampling Soils for Highway Subgrades (D 420 – 45) was passed by letter ballot of the committee during the year. Section 2C of this subcommittee is now drafting a Proposed Method on Borings and Sampling for Soil Investigations of Embankment Materials. The subcommittee is now considering what course it should take in regard to setting up methods for exploration. It is discussing the value of setting up definition of terms used in soil investigations.

Subcommittee on R-3 on Physical Characteristics of Soil (H. Allen, chairman) held a meeting in June, 1949. The organization of the subcommittee was completed during the year and the three sectional committees (A, B, and C) are functioning satisfactorily. During the year, Section A has prepared revisions of Tentative Method of Test for Specific Gravity of Soils (D 854 – 45 T). Section A has also prepared a Proposed Method of Test for Amount of Material Finer than No. 200 Sieve for Soil. As a third item the section has recommended the incorporation of tentative revisions[7] into the Standard Method of Mechanical Analysis of Soils (D 422 – 39). These items have been submitted for letter ballot to Subcommittee R-3 and have been approved. Section B on Atterberg Limits has prepared revisions of the Method of Test for Liquid Limit of Soils (D 423 –39) and Method of Test for Plastic Limit and Plasticity Index of Soils (D 424 – 39) and will submit them to the subcommittee members during the next Annual Meeting. The revision of these methods should be completed during the next year. Section C on Moisture Content and Density, has prepared a questionnaire, by means of which it was hoped to develop desirable changes in Method of Test for Moisture-Density Relations of Soils (D 698 – 42 T). This section also gave consideration to the revision of a Proposed Method of Test for the Field Determination of Density of Soil In Place.

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[8] The letter ballot vote on these recommendations was favorable; the results of vote are on record at ASTM Headquarters.

Subcommittee R-5 on Structural Properties of Soil (D. M. Burmister, chairman). —Sections A and C of this subcommittee held meetings during June, 1949. Section A on Consolidation has continued a program of research and study in order to obtain basic information on consolidation for the purpose of setting up reasonable and practical requirements and procedures for the consolidation test. No effective work was done by Section B on Direct Shear during the year. The chairman of this sectional committee is planning on compiling several articles relating to direct shear tests which have been printed during the past year. These articles will be submitted to sectional committee members for their information and for discussion. Section C on Methods of Testing Soils Under Triaxial Loading is sponsoring a symposium on Testing Soils Under Triaxial Loading which will be presented at the 1950 Annual Meeting.

Subcommittee R-6 on Physico-Chemical Properties of Soil (E. F. Preece, chairman).—This subcommittee is now completely organized and functioning. At present, attention is being given only to the identification and measurement of physico-chemical properties. Significance and application will be the subject of a later phase of the work after the present work is much more advanced. In order to take advantage of the work that has been done, the membership of the subcommittee was selected to include authorities from those fields in which present knowledge was developed. Membership includes persons familiar with the agricultural, ceramic, engineering, instrumentation, physical chemistry, and mineralogy phases of the work. The program for the coming year is concerned with the development of the procedures for the determination of the constants of composition, the initial phase of the broader subject of procedures for the determination of the constants of identification. Coordinated assignments have been made for the following procedures: differential thermal analyses, spectroscopic analyses, spectrographic analyses, petrographic analyses, chemical and X-ray diffraction analyses, and cationic exchange capacity determination.

Subcommittee R-7 on Identification and Classification of Soils (G. W. McAlpin, chairman).—This subcommittee held meetings during June and October of 1949. The subcommittee is acting as a committee for the preparation of a symposium at the 1950 Annual Meeting on the subject of Identification and Classification of Soils. The committee is to give consideration, after the Annual Meeting has been held, to the preparation of a written symposium on the above subject which will incorporate the papers presented at the meeting, together with other papers necessary to give coverage to this important subject. The subcommittee is working toward the preparation of a tentative standard on soil nomenclature with respect to grain size.

Subcommittee R-8 on Special and Construction Control Tests (H. F. Clemmer, chairman).—The various sectional committees of R-8 have held meetings with a view to completing their organization and planning a program of work. The organization of these sections is now complete and several of the sections have indicated progress in formulating a program of research to be carried on. Section D-1 of this subcommittee reported that the organization of their membership was completed during the year and that all soil cement standard testing procedures were reviewed and found to be in order.

Subcommittee R-9 on Dynamic Properties of Soil (R. K. Bernhard, chairman).—One meeting was held in June, 1950, during the Annual Meeting, where the scope outline was accepted. The scope is as follows: "The scope of this subcommittee includes compilation, study, and correlation of data pertaining to dynamic properties of soils. Characteristic dynamic properties of soils to be studied are: elasticity, damping, standing and propagation waves, consolidation, settlement, densification, bedding values, etc. under dynamic loads, with or without static surcharges, and resonance phenomena of the vibratory systems. Methods to determine these dynamic properties are to be compared". The following research projects are under discussion: (*1*) A Joint Highway Research Project, sponsored by the State Highway Department of New Jersey, the Bureau of Public Roads, and Rutgers University, referring, in particular, to the measurement of wave propogation and phase velocities in connection with the determination of the Young-Modulus, Shear-Modulus, and Bulk-Modulus of soils, and to pilot tests to determine soil densities by means of radio-active cobalt; (*2*) A research project, sponsored by the Rensselaer Polytechnic Inst. on soil density determination by means of X-rays; (*3*) Experiments with ultrasonic waves (pulse technique) in soils, referring to their propagation velocities; and (*4*) Tests with pressure cells, and the measurement of pile friction started by the Association of American Railroads.

Subcommittee R-10 on Bearing Tests of Soil In Place (L. A. Palmer, chairman of Section A; and A. E. Cummings, chairman of Section B).—*Section A.*—This section held a meeting during June, 1949. Three separate loading test procedures were submitted by the sectional committee for the A.S.T.M. Book on Testing of Soils.

Section B.—This section held a meeting during June, 1949. Their work on a proposed tentative standard, A Method of Test to Determine the Load-Settlement Relationship for Individual Piles, has been submitted to the committee for action.

Joint Subcommittee of Committee D-4 and D-18 on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman).—There was no scheduled meeting of the joint committee during the year. The status of the specifications under the jurisdiction of the joint committee has been the subject of correspondence and the following recommendations made: It is recommended that the Method of Test for Shear Strength of Flexible Road Surfaces and Fills by the Burggraf Shear Apparatus, (D 916 - 47 T) remain as tentative. Research on this method is still in progress and it is believed that it should remain as tentative until this research is completed. Method of Testing Soil-Bituminous Mixtures (D 915 - 47 T) was considered by Section D-2 of Subcommittee R-8 of Committee D-18 and it was decided to determine by cooperative field and laboratory investigations whether or not this method is suitable, as now written, for use as a referee test method. It is recommended that this method remain as tentative until this research is completed. Specifications for Materials for Stabilized Base Course (D 556 - 40 T) and Specifications for Materials for Stabilized Surface Course (D 557 - 40 T), respectively, are being re-submitted to the committee for further study in order to correlate them with the latest A.A.S.H.O. specifications. It is recommended that these specifications remain as tentative until a report as to any recommended changes may be received.

Subcommittee S-1 on Specifications and Methods of Tests (F. J. Converse, chairman).—As individual standard subcommittees were not formed during the year to handle action on pending standards, this work was handled through the appropriate R subcommittee chairmen.

This report has been submitted to letter ballot of the committee which consists of 92 voting members; 70 members returned their ballots, of whom 64 have voted affirmatively and 1 voted negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

R. F. BLANKS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Test for Amount of Material Finer Than No. 200 Sieve for Soil (D 1140 - 50 T).

Revision of Tentative Method of:

Test for Specific Gravity of Soils (D 854 - 45 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives appear in the 1950 Supplement to Book of ASTM Standards, Part 3.

On December 14, 1950, the Administrative Committee on Standards accepted the recommendation of Committee D-18 that the Method of Test to Determine the Load Settlement Relationship for Individual Piles be published as tentative. The new tentative method appears in the 1950 Supplement to Book of ASTM Standards, Part 3, bearing the designation D 1143 - 50 T.

REPORT OF COMMITTEE D-19

ON

INDUSTRIAL WATER[1]

Committee D-19 on Industrial Water and its subcommittees held regular meetings in Atlantic City, N. J., June 30 and July 1, 1949, and in Pittsburgh, Pa., March 2 and 3, 1950. Subcommittee VII on Water-borne Industrial Waste sponsored a Round Table Discussion on "The Need for Standards for the Examination of Water-borne Waste," presented during the Session on Water, June 30, 1949.[2]

All of the methods under the jurisdiction of Committee D-19 as of December 1949, while available in Part 5 of the 1949 Book of A.S.T.M. Standards, have also been published for convenient use in a desk-size, paper-bound volume under the title of "A.S.T.M. Standard Methods on Water," December, 1949.

The Executive Committee of the Joint Research Committee on Boiler Feedwater Studies, on which A.S.T.M. is represented as a sponsoring organization, held a meeting during the Annual Sessions of The American Society for Mechanical Engineers in New York, N. Y. on December 1, 1949. With the reorganization of the committee two years ago it became the policy of the committee to elect a new chairman and reconsider officers every two years. S. F. Whirl, formerly first vice-chairman, was chosen chairman. R. W. Seniff was advanced to first vice-chairman and E. P. Partridge was elected second vice-chairman. R. C. Ulmer was appointed secretary.

The chairmen of the three subcommittees previously organized are: *Deposits*, R. C. Bardwell; *Steam Contamination*, W. L. Webb; and *Corrosion*, J. W. Ryznar.

During 1949, the Joint Research Committee co-sponsored sessions on water problems at the A.S.M.E. Semi-Annual Meeting in San Francisco; the Tenth Annual Water Conference, Engineers' Society of Western Pennsylvania, in Pittsburgh; and at the annual meeting of the A.S.M.E. in New York. The committee is continuing its plans for the sponsoring of several sessions during the present year. The three subcommittees met at the conclusion of the Executive Committee meeting and formulated plans for completion of their organization and outlined their objectives.

The A.S.T.M. is represented on the Subcommittee on Care of Steam Boilers and Other Pressure Vessels in Service of the A.S.M.E. Boiler Code Committee. At an open meeting of this subcommittee, during the annual meeting of the A.S.M.E. on December 1, 1949, attended by members of both the chemical and mechanical sections, the chemical section approved without change the fifth draft of the "Proposed Revisions and Addenda to Suggested Rules for the Care of Power Boilers, Section VII of A.S.M.E. Boiler Construction Code." This fifth draft contained a number of revisions made by the working group on the basis of suggestions received after the fourth draft was published as information in *Mechanical Engineering*, June, 1948.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The discussion appears in the ASTM BULLETIN, No. 162, December, 1949, and includes the following: Introduction, L. K. Herndon, p. 35; "Analysis of Water-borne Industrial Waste: The Need for Uniformity in Methods of Analysis and Reporting," G. D. Beal and S. A. Braley, pp. 36, 37; "Gaging and Sampling Water-borne Industrial Waste," C. F. Hauck, pp. 37 to 43.

The chemical section voted to submit the fifth draft to the Boiler Code Committee for approval not later than the annual meeting of the A.S.M.E. in 1950. In the meantime, an attempt will be made to adjust some differences of opinion expressed by members of the two sections.

TABLE I.—DATA ON THE EFFECT OF IMPURITIES ON THE PROPOSED A.S.T.M. NON-REFEREE METHOD OF TEST FOR CARBON DIOXIDE IN INDUSTRIAL WATERS.

Sample	Total CO_2 present, ppm.	Known Amounts of Impurities, ppm.						Total CO_2 Found, ppm.
		PO_4	SiO_2	Fe	Al	Mg	NH_4	
A	20.5	...	...	...	...	...	...	21.0 (avg. 10 samples)
B	26.0	...	...	...	...	...	...	27.0 (avg. 10 samples)
C	46.5	...	...	...	...	...	...	46.5 (avg. 10 samples)
D	9.0	...	...	...	...	...	...	9.0, 9.5, 9.5, 8.0 and 11.0
E	10.5	...	...	...	...	...	...	9.5, 11.0, 10.0
F	59.0	200	...	...	...	...	...	56
G	52.0	200	...	...	...	...	...	53, 52, 50, 52, 51.5
H	52.0	...	50	...	...	...	...	55, 53, 51, 52
I	52.0	...	...	...	25	...	...	56, 53, 54, 53
J	52.0	...	...	50	...	...	...	51, 52, 54, 49, 51.5
K[a]	52.0	...	...	...	...	25	...	55, 54, 53, 52
L	52.0	...	...	...	...	...	...	51.5, 51.5
M	10.5	200	...	...	...	...	...	11.5, 11.0
N	10.5	...	30	...	...	...	...	9.0, 10.0
O	10.5	...	...	50	...	...	...	9.5, 10.0
P	10.5	...	...	...	25	...	...	8.5, 9.5
Q	10.5	...	...	...	...	25	...	12.0, 10.0
R	47	...	...	...	...	...	15	46.5 (3 determinations)

[a] The first titration with 0.04 *N* HCl has to be carried out until the solution remains colorless for 30 sec. Roughly speaking about 10 min. is required with 25 ppm. Mg.

At present there are 99 members serving on Committee D-19, of whom 38 are classified as producers, 39 as consumers, and 22 as general interest members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, Max Hecht.

First Vice-Chairman, Robert C. Adams.

Second Vice-Chairman, F. R. Owens.

Secretary, Robert T. Sheen.

NEW TENTATIVES

The committee recommends for publication as tentative the following four methods, as appended hereto:[3]

Tentative Method of Test for Sodium and Potassium in Industrial Water.—This method is a referee type method and describes a gravimetric procedure for the determination of sodium and potassium in industrial water.

TABLE II.—EXPERIMENTAL DATA ON REPRODUCIBILITY OF THE PROPOSED REVISED A.S.T.M. METHODS FOR SILICA (D 859).

REFEREE METHOD USING PERCHLORIC ACID, FIVE DETERMINATIONS OF KNOWN SOLUTION WITH 0.0122 g. SiO_2

Value	SiO_2, g.		
	Pyrex Beaker	Porcelain Dish	Platinum Dish
Mean	0.0123	0.0115	0.0128
Range	0.0004	0.0011	0.0010

REFEREE METHOD USING HYDROCHLORIC ACID, NINE DETERMINATIONS OF KNOWN SOLUTION WITH 0.0122 g. SiO_2

Value	SiO_2, g.
	Porcelain Dish
Mean	0.0103
Range	0.0010

NONREFEREE METHODS WITH KNOWN SILICA SOLUTIONS

Known SiO_2 Content, ppm.	Range of five Determinations on Separate Samples, ppm.	
	Nonreferee Method A G.E. Spectrophotometer at 440 mμ.	Nonreferee Method B Beckman Spectrophotometer at 410 mμ
2	0.05	...
3	...	0.03
6	...	0.05
9	...	0.13
12	...	0.22
20	0.70	...
50	0.80	...

Tentative Method of Test for Electrical Conductivity of Industrial Water.—This method is a referee type method for the measurement of ionized substances in water and is useful as a relative measure of dissolved solids in water.

Tentative Method of Test of Hardness in Industrial Water.—This method is a soap titration procedure, one of several non-referee methods being prepared by the committee.

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[3] These new methods were accepted as tentative by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

Method for the Identification of Types of Microorganisms.—This procedure describes the application of the biological microscope. The procedure was first published as information in the 1948 annual report of the committee.

REVISIONS OF TENTATIVES

The committee recommends that the following three tentatives be revised as indicated and continued as tentative:

Tentative Methods of Test of Total Carbon Dioxide, and Calculation of the Carbonate and Bicarbonate Ions in Industrial Water (**D 513 - 48 T**).[4]— The committee recommends the addition of the Strontium Chloride Method as a non-referee procedure, appended hereto.[5] See Table I for supporting data.

Tentative Methods of Test for Silica in Industrial Water (**D 859 - 47 T**).[4]—The revised methods, appended hereto,[5] represent a revision of both substance and form of the present tentative. See Table II for supporting data.

Tentative Recommended Practice for Application of X-Ray Diffraction Methods To Water-Formed Deposits (**D 934 - 47 T**).[4]—The revised method, appended hereto,[5] represents a complete revision of both substance and form.

ADOPTION OF TENTATIVE AS STANDARD

The committee recommends that the following tentative be approved for reference to letter ballot of the Society for adoption as standard, with revision as indicated:

Tentative Method of Test for Total Aluminum and Aluminum Ion in Industrial Water (**D 857 - 48 T**):[4]

Section 2(b).—Add the following sentence: "Fluorine up to 0.0526 epm (1 ppm. as F.) does not interfere; at 0.2631 epm (5 ppm. as F.) a slight amount of interference may be encountered giving slightly higher results, particularly when the color is measured photometrically."

NEW STANDARD

The committee recommends that the Definitions of Terms Relating to Industrial Water, appended hereto,[6] be referred to letter ballot action of the Society for immediate adoption as standard. For greater convenience in use the committee has assembled various definitions under its jurisdiction, previously scattered throughout several methods on Industrial Water, into one document. A few additional definitions have been added, but because nearly all of the definitions have already been adopted as standard as part of the various methods, the committee recommends the immediate adoption of the proposed Standard Definitions of Terms Relating to Industrial Water and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the revision of the *Standard Method of Reporting Results of Examination and Analysis of Water-formed Deposits* (**D 933 - 49**),[4] expanding the method to include the reporting of chemical analysis and the results of examination by petrographic microscope and X-ray diffraction. The revised method is appended hereto,[7] and the committee asks for a nine-tenths affirmative vote at the Annual Meeting in order that this recommendation may be referred to letter ballot of the Society.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the

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[4] 1949 Book of A.S.T.M. Standards, Part 5.

[5] The revised methods were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[6] The new standard definitions were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 5.

[7] The revised method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 5.

following four tentatives be continued as tentative without revision, pending further study:

Tentative Method of:

Sampling Boiler Water from Stationary Boilers (D 860 - 48 T)[4]

Tentative Method of Test for:

Iron Bacteria in Industrial Water (D 932 - 47 T)[4]
Nitrate Ion in Industrial Water (992 - 48 T)[4]
Sulfate-Reducing Bacteria in Industrial Water and Water-formed Deposits (D 993 - 48 T).[4]

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[8]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Advisory (Max Hecht, chairman).—held meetings on June 30 and October 16, 1949, and March 3, 1950.

It approved the election of new members to the committee, the appointment of Claude K. Rice, as chairman of Subcommittee II, Editorial, and the report of the Nominating Committee, consisting of L. D. Betz, chairman, Orrin M. Elliott and Roy F. Weston, for nominations of officers to serve the two-year term starting July 1, 1950.

It approved the organization of new sub-sections and the scopes for new proposed methods.

Two members of the subcommittee tendered resignations: E. P. Partridge, as chairman of Subcommittee II, Editorial, effective June 30, 1949, and R. E. Hall, as secretary of the committee, effective April 1, 1950. Appropriate citations have been recorded in the minutes of the committee in recognization of the valuable services rendered by these two members.

The officers sent to the family of the late Carter S. Cole, and to the Society, letters of condolence on his death in November, 1949. Mr. Cole, by virtue of his knowledge and judgment, served the committee as one of the Society advisors.

A simplified policy was approved by the subcommittee for the processing of proposed methods and revisions through the committee. The following procedure is effective: When the substance of a proposed method or proposed revisions, are agreed upon by the section or subsection, approvals will be made by the subcommittee chairman for substance, by the committee chairman for policy, and by Subcommittee II chairman for editorial form.

When the subject matter has not been discussed at an earlier regular subcommittee meeting, the subcommittee chairman may release drafts of the material to the subcommittee membership for comment and suggestions, but not necessarily for letter ballot vote. When the subject matter has been discussed at a subcommittee meeting, the subcommittee chairman may release the material in draft form to the entire membership of the committee for comment, suggestions, and letter ballot vote; the vote is to be interpreted as a concurrent action of the subcommittee and the committee. The material then is reviewed at a regular meeting of the subcommittee and of the committee, prior to offering the necessary recommendation from the committee to the Society for publication and adoption.

Subcommittee II, Editorial (Claude K. Rice, chairman).—This subcommittee, held no meetings during the year. See report of Subcommittee I, Advisory, for change in chairman.

Section A on A.S.T.M. Manual on Industrial Water held meetings June 29, 1949, and March 2, 1950. Drafts of seven of the nine chapters[9] have been completed. J. A. Holmes was appointed to the Section to prepare Chapter III on "Difficulties Caused by Water in Industry," and A. K. Light was appointed to prepare Chapter VIII on "Analysis of Water-Formed Deposits." The Section has scheduled the completion of these two chapters for June, 1950, and the complete editing of the Manual by December, 1950. The manuscripts will be submitted to the Society with the recommendation for publication early in 1951.

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[8] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[9] *Proceedings*, Am. Soc. Testing Mats., Vol. 47, p. 412 (1947).

Subcommittee III on Methods of Sampling (Orrin M. Elliott, chairman).—The subcommittee held meetings on June 30, 1949, and March 2, 1950. It recommended the continuation as tentative of Method for Sampling Boiler Water from Stationary Boilers, D 860 T, pending revision of substance.

Current projects in Section A on Sampling of Water include the preparation in draft form of a proposed method for Sampling of Water at Subatmospheric Pressure. The draft is currently being studied by the subsection. The second project is that of preparing a Proposed Specification for Sampling Equipment. The subsection has been enlarged, and a new chairman, R. A. Lorenzini, has assumed the project. The specification is to include all necessary equipment for the sampling of water, and of steam. An exhaustive study is being made to evaluate all of the items that are to be included in the proposed specification, and these include, among others, chemical composition and mechanical properties of readily available metals and alloys suitable for sampling, nozzles, lines, valves, fittings, and cooling coils, and weldability of such materials. Nominal designs of cooling coils will be developed.

Area of heat transfer surfaces and the amount of cooling water required will be supplied in the form of curves.

Section C on Sampling of Steam reported no revision in the Tentative Method of Sampling Steam (D 1066 – 49 T) and is awaiting the results of operating tests of the sampling nozzles. The chairman, B. J. Cross, reports that he attended a meeting of the A.S.M.E. Boiler Test Code Committee, and submitted a copy of the above Tentative for study by that group. He further reports that the American Boiler and 'Affiliated Industries' Standards Committee has prepared a Fair Practice Code in which is incorporated methods for determining Steam purity, and that this organization has included the following methods in the Code: Sampling of Steam (1066 T), Method of Test for Suspended and Dissolved Solids in Industrial Water, (1069 T), Methods of Test for Total Carbon Dioxide, etc. (D 513 T), and the proposed Tentative Method of Test for Electrical Conductivity of Water, appended to this report.[10]

Subcommittee IV on Methods of Analysis (Frank E. Clarke, chairman).—The subcommittee held regular meetings on July 1, 1949, and March 3, 1950. It completed work on various methods and revisions of existing methods, reported elsewhere in this report, and recommended them for transmission to the Society for publication and adoption.

Section A on Analytical Methods is proceeding with proposed revisions to methods of Test for Orthophosphate, etc. (D 515) and Methods of Test for Dissolved Oxygen in Industrial Water (D 888 T). Progress is further reported on the methods for the following determinations, not as yet supplied to the subcommittee in final draft form: copper; clarity, turbidity, and color; chlorine; ammonia; fluoride; and matter extractable with chloroform. The proposed methods for clarity, turbidity, and color will be based on the light scattering effect (Tyndall effect), light image distortion, and spectrophotometric properties, respectively. The following four new projects were authorized: determination of hydrogen, hydrogen sulfide, bromide and iodide, and preparation of a master scheme for the analysis of all constituents in industrial water.

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[10] The new method was accepted by the Society as tentative and appears in the 1950 Supplement to ASTM Standards, Part 5.

Section B on Electrometric Methods, with the completion of the Method of Electrical Conductivity in Water, which is appended[10], will survey the requirements for procedures using electrometric methods.

Section C on Special Methods has completed the preparation of two methods: Microorganisms and X-ray Diffraction (D 934 T) both of which are appended hereto.[11]

The section adopted rules for naming of compounds, published as a revision to Method of Reporting Analysis of Water-formed Deposits (D 933), appended to this report.[11]

Progress is reported on current projects of this section, including proposed methods for the flame photometer (a non-referee procedure for sodium and potassium); visual procedures for the spectroscope; and changing of the subsection on Petrographic Microscope to Chemical Microscope, with change in scope to permit the use of chemical microscope for the study of deposits.

Section D on Chemical Analysis of Water-formed Deposits reports the completion of an outline, preparatory to the insertion of necessary procedures for the complete chemical analysis of water-formed deposits.

Subcommittee V on Classification, (S. K. Love, chairman).—The subcommittee held regular meetings on June 30, 1949, and March 2, 1950. It completed work on various methods, appended.

The subsection on the preparation of a proposed specification for reagent-grade water was instructed to prepare specifications for: (*a*) use in procedures and processes where highest quality water is required, and (*b*) general purposes.

Section B on Nomenclature and Terminology reviewed and approved various definitions submitted to it and organized the proposed Tentative Definitions of Terms Relating to Industrial Water, appended to the report.[6]

Subcommittee VI on Methods of Testing (V. V. Kendall, chairman).—The subcommittee held regular meetings on June 30, 1949, and March 2, 1950.

The subcommittee completed a preliminary draft of a Proposed Specification for Substitute Ocean Water, approved by the subcommittee and the committee. The draft has been submitted to the Advisory Committee on Corrosion for disposition. That committee had requested Committee D-19 to prepare and submit a simplified formula.

A series of specifications covering service testing methods for tubular materials for various types of equipment and applications is in preparation. Development of a program for the future work of the subcommittee is now in progress.

Subcommittee VII on Water-Borne Industrial Wastes (L. D. Betz, chairman).—The subcommittee held regular meetings on June 30, 1949, and March 2, 1950.

Section A on The Critical Constituents of Water-Borne Industrial Wastes, whose scope is the selection of key determinations on a specific waste by which it may be evaluated, has prepared a rather complete outline of industries contributing water-borne wastes. It is hoped that each industry may be classified as to the character of its wastes and that a list will be prepared of the important and key determinations that should be made on a waste from a particular industry so that the character of the waste may be evaluated.

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[11] The revised method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 5.

Section B on Methods of Analysis for Industrial Waste has organized 9 subsections, each charged to examine carefully the best existing methods of analysis and to develop and prepare a proposed method. The methods of analyses under investigation at the moment are: suspended solids; turbidity and color; dissolved solids; acidity, alkalinity and pH; oxygen demand; taste and odor; chlorine demand; scum forming constituents; and toxicity to aquatic life. Three additional subsections, one on phenol, one on sulfur compounds, and another on cyanides are in the process of formation. Subcommittee IV has prepared methods on industrial water for several of these determinations. Such procedures will first be studied to determine if suitable changes may be incorporated in the existing method of analysis to make it suitable for waterborne industrial waste.

Section C on Methods of Sampling, Gaging, and Preservation of Samples has completed organization of two subsections. One subsection will study methods of sampling and gaging industrial waste waters and the other will investigate methods of preservation of samples.

Section D on Methods of Reporting Results of Analysis is being organized into four groups. The first group will study the reporting of analysis having to do with oxygen demand, acidity, and alkalinity of such wastes as pickle liquor, alkaline cleaning wastes, plating wastes, etc. The second group will cover the reporting of metallic and nonmetallic ions in water. The third group will cover the gaseous substances such as O, CO_2, SO_2, H_2S, etc. The fourth group will study the reporting of contaminants such as cyanides, chromates, phenols, oils, greases and other organic matter.

This report has been submitted to letter ballot of the committee, which consists of 99 members; $70\frac{1}{2}$ members returned their ballots, of whom $65\frac{1}{2}$ have voted affirmatively and 4 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the recommendation that the Specifications for Substitute Ocean Water be published as tentative. This recommendation was accepted by the Standards Committee on September 26, 1950, and the new tentative specifications appear in the 1950 Supplement to Book of ASTM Standards, Part 5, bearing the designation D 1141 – 50 T.

REPORT OF JOINT RESEARCH COMMITTEE
ON
BOILER FEEDWATER STUDIES[1]

Sponsored by:
Am. Boiler Manufacturers Assn. and Affiliated Industries,
Am. Railway Engineering Assn.,
Am. Soc. Testing Mats.,
Am. Soc. for Mechanical Engrs.,
Am. Water Works Assn., and
Edison Electric Inst.

During 1949, the activities of the Joint Research Committee on Boiler Feedwater Studies were concerned chiefly with sponsorship of technical papers at various society meetings, organization of subcommittee personnel, and planning of subcommittee work for the future.

At the Semi-Annual Meeting of the American Society of Mechanical Engineers in San Francisco in June, a program was co-sponsored which consisted of five papers dealing with water problems and practices on the West Coast and a research study on quality of steam condensate as related to sodium sulfite in the boiler water. In October at the Water Conference of the Engineers' Society of Western Pennsylvania in Pittsburgh, the co-sponsored program consisted of four papers on water problems in starting a new station, problems of generating pure steam at high pressures, and deaeration of feedwater. Four papers were presented at the co-sponsored session at the Annual A.S.M.E. Meeting in New York in December pertaining to hide-out of sodium phosphate in high-pressure boilers, make-up for high-pressure by-product steam plants, sulfite and silica in boiler water, and an automatic degasser for steam samples.

The Joint Research Committee met in December during the Annual A.S.M.E. Meeting in New York. In line with committee policy, Chairman W. C. Schroeder retired to ex-officio status; the first vice-chairman and the second vice-chairman advanced; a new second vice-chairman was elected, and a new secretary appointed to replace T. H. Daugherty, who resigned. The officers and subcommittee chairmen are now as follows:

Chairman.—S. F. Whirl, Chemical Operating Engineer, Power Stations Dept., Dusquene Light Company, Pittsburgh, Pa.

1st Vice-Chairman.—R. W. Seniff, Baltimore and Ohio Railroad Co., Pratt and Arlington Avenues, Baltimore, Md.

2nd Vice-Chairman.—E. P. Partridge, Director, Hall Laboratories, Inc., Pittsburgh, Pa.

Secretary.—R. C. Ulmer, E. F. Drew & Co., Inc. 15 E. 26th St., New York, N. Y.

Subcommittee on Deposits (*Chairman*).—R. C. Bardwell, Supt. of Water Supply, Chesapeake & Ohio Ry. Co., Richmond, Va.

Subcommittee on Corrosion (*Chairman*).—J. W. Ryznar, Technical Director, Nat. Aluminate Corp., Chicago, Ill.

Subcommittee on Steam Contamination (*Chairman*).—W. L. Webb, Am. Gas & Elec. Service Co., New York, N. Y.

Respectfully submitted on behalf of the committee,

S. F. WHIRL,
Chairman.

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.**

REPORT OF COMMITTEE D-20
ON
PLASTICS[1]

Committee D-20 on Plastics and its subcommittees held three meetings during the year: in Atlantic City, N. J., on June 29 and 30, 1949; in New York, N. Y., on October 31 and November 1, 1949, and in Old Point Comfort, Va., on March 21 and 22, 1950.

The committee has approved amendments to its Regulations to provide for a membership secretary, defining his duties of maintaining a complete membership list, as well as allowing the retiring chairman to serve on the Advisory Committee.

The committee has under consideration the matter of participation in a Committee on Plastics being set up under the International Standards Organization.

J. L. Williams was appointed chairman of Subcommittee IX to succeed L. A. Sontag, who resigned because of other duties. Committee D-20 held its election of officers at its March meeting with the following being elected for a two-year term, taking office at the close of the Annual Meeting in June, 1950:

Chairman, G. M. Kline.
First Vice-Chairman, H. W. Paine.
Second Vice-Chairman, R. K. Witt.
General Secretary, B. L. Lewis.
Membership Secretary, T. J. Dietz.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendation that the Recommended Practice for Describing Form of Specimen and Direction of Testing Plastics be published as tentative. This recommendation was accepted by the Standards Committee on September 14, 1949, and the new tentative recommended practice appears in the 1949 Book of A.S.T.M. Standards, Part 6.

Revisions of the Tentative Specifications for Nonrigid Vinyl Chloride Plastics (D 744 - 44 T) were accepted by the Standards Committee on January 17, 1950. The revised tentative specifications appear in the 1949 Book of A.S.T.M. Standards, Part 6.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication one new tentative and is recommending the revision of two tentatives, adoption as standard of 14 tentatives with revisions in one of them, revision of two standards for immediate adoption, and the withdrawal of one tentative. The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.

The committee has considered all of the other tentatives, not mentioned in the Appendix, which are under its jurisdiction and which have been issued for two years or longer and recommends that they be continued in their present status as further revisions in them are contemplated.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

ACTIVITIES OF SUBCOMMITTEES

This year has found all the subcommittees very active. The Subcommittees on Optical Properties and on Specifications, as well as on Analytical Methods, have expanded their work. Subcommittee XIV on Conditioning, which was formerly known as the Joint Subcommittee on Conditioning of Committee D-9 and D-20, changed its designation to avoid confusion and for uniformity of title to conform with the same designation within Committee D-9.

Subcommittee I on Strength Properties (M. E. Marks, chairman).—During the past year, the Section on Tensile and Compressive Strength has been active in revision of its methods. The Impact Strength Section is now studying the falling-ball method. Revision of the fatigue method is now under way, while the Section on Shear is studying a double shear method. The Section on Flexural Strength is developing revisions to its present method, as well as working on the determination of flexural strength of thin sheets.

The Bonding Strength Section is working on a new type of adhesive so that bonding strengths can be determined on other plastics such as silicone materials. Work is also progressing on a method for bursting strength of tubes in the Section on Bursting Strength. The Standard Method of Long-Time Tension Tests of Plastics (D 674 - 48) is being revised and will be submitted to the subcommittee. The Section on Dynamic Stress, which has just recently been organized, is pursuing this problem.

Subcommittee II on Hardness (R. B. Rice, chairman).—The Section on Scratch Resistance has been set up to study further a method of test, and some preliminary work has been done. The Method of Test for Resistance of Transparent Plastics to Surface Abrasion (D 1044 - 49 T) has now been in use for the past year, but round-robin studies are still being intensively pursued in different laboratories to establish a firmer basis on which the accuracy and the reproducibility of the tests can be obtained.

The Section on Abrasion Resistance (Mechanical Effects) is carrying out extensive work on a method, and two detailed progress reports have been submitted by the chairman of this section at previous meetings. This work will be continued and other equipment will be tested on a comparison basis.

Subcommittee III on Thermal Properties (E. B. Cooper, chairman).—The Section on Deformation Under Load is preparing a test method, particularly applicable to polyethylene. The Section on Flammability is running round-robin tests on flammability equipment in cooperation with a series of laboratories with a new method which is simpler to control and calibrate. Low-temperature brittleness tests are still continuing, and round-robin tests are being conducted on several types of machines at this time.

Subcommittee IV on Optical Properties (G. W. Ingle, chairman).—The subcommittee is actively reviewing the possibilities of using the Method of Test for 60-deg. Specular Gloss of Paint Finishes (D 523 - 48 T) to see if it is applicable to plastic materials. The Method of Test for Diffusion of Light by Plastics (D 636 - 43) is being studied and will be revised within the next year.

Subcommittee V on Permanence Properties (W. C. Goggin, chairman) has been quite busy this past year. The study to be made for mounting films and samples for exposure to light has been discontinued, since commercial fixtures are available.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The Method of Test for Water Vapor Permeability of Plastic Sheets (D 697 - 42 T) is being retained as tentative, awaiting completion of cooperative work with Committee E-1 on Methods of Testing in an attempt to coordinate tests on this subject.

Further work is being carried on to revise the Method of Test for Resistance of Plastics to Chemical Reagents (D 543 - 43).

A method of measuring warpage of plastic sheets is in preparation.

A modification of the Cartwright Method of determining gas permeability of plastic films will soon be proposed.

Subcommittee VI on Specifications (F. W. Reinhart, chairman), has prepared and revised a number of specifications.

Revisions of a number of specifications are under way, including those for: Cellulose Acetate Molding Compounds (D 706 - 48 T), for Cellulose Acetate Butyrate Molding Compounds (D 707 - 47 T), for Nonrigid Vinyl Chloride Acetate Resin Plastics (D 742 - 46 T), and Nylon Injection Molding Compounds (D 789 - 44 T).

New tentative specifications for decorative plastic laminates, as well as for various plasticizers, are being prepared. Work is continuing on polyethylene, vinyl resins, cellulose propionate plastics, and alkyd molding compounds.

The Tentative Specifications for Nonrigid Vinyl Butyral Plastics (D 745 - 44 T) are being withdrawn.

Subcommittee VII on Analytical Methods (D. E. Northrop, chairman) has been quite active during the past year. At the present time, test methods for vinyl chloride polymers and copolymers, as well as procedures for the determination of plasticizer content of cellulose ester plastics, are under consideration. Work is continuing on other methods of testing plasticizers to be added to Tentative D 1045 - 49 T.

Subcommittee VIII on Research (J. D. Ryan, chairman) is continuing its work on the fundamental aspect of impact and flow properties, as well as on the degradation of plastics.

The committee is continuing its function of assisting educational institutions in an advisory capacity on research problems suitable for investigation by graduate students.

At the November, 1949, meeting of the subcommittee the following four papers were presented:

"The Use of Electrical Measurements in the Study of Physical Properties of Plasticized Polyvinyl Resins," by A. J. Warner,[3]
"An Investigation of the Fatigue Behavior of Polystyrene," by J. F. Throop,
"Some Fundamentals of the Injection Molding Process," by G. D. Gilmore,
"Some Instruments for Measuring the Dynamic Mechanical Properties of Plastic Materials,"[4] by L. E. Nielsen.

At the March, 1950, meeting of the subcommittee the following three papers were presented:

"Characteristics of Carbon Arc Light Sources for Accelerated Exposure Testing," by W. W. Lozier,
"Some Rapid Approximate Statistical Procedures," by Frank Wilcoxon,
"The Mechanism of the Degradation of Polyamides," by Frank W. Reinhart.

Papers of an original nature are solicited by the committee for presentation at future meetings.

Subcommittee IX on Molds and Molding Processes (J. L. Williams, chairman) has been revising some of its methods and also has been working on mold procedures and molds for thermosetting materials, as well as thermoplastic molding materials. Work is continuing in the subcommittee on the design of a compression mold for making phenolic tensile specimens.

This subcommittee prepared the new

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[3] ASTM BULLETIN, No. 165, April, 1950, p. 53 (TP79).

[4] ASTM BULLETIN, No. 165, April, 1950, p. 48 (TP74).

Tentative Recommended Practice for Injection Molding of Specimens, of Thermoplastic Materials appended hereto.[5] Work has been initiated during the past year which includes a new apparent density and bulk factor test for granular plastic materials.

Subcommittee X on Definitions, Nomenclature, and Significance of Test (G. W. Clark, chairman) is still continuing its work on definitions of sheet, continuous sheet, and film. The following terms are being reconsidered by the subcommittee: "thermoset" noun; as well as "thermoelastic" and "quasi-thermoelastic."

The Section on Significance of Tests will now be entitled Correlation of Terms and Definition. This section will handle correlation with the special committee appointed by Committee E-1 and will correlate definitions within Committee D-20 and between Committee D-20 and other technical committees of the Society.

The Tentative Descriptive Nomenclature of Objects Made from Plastics (D 675 - 45) is under revision and the changes will include pictures for illustrating the visible characteristics of a plastic object.

This subcommittee functions jointly for Committees D-9 and D-20.

This report has been submitted to letter ballot of the committee, which consists of 168 members; 101 members returned their ballots, of whom 91 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

G. M. KLINE,
Chairman.

B. L. LEWIS,
Secretary.

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[5] The new recommended practice was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 6.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON PLASTICS

In this Appendix are given a list of one new tentative, proposed revisions in two standards and of two tentatives covering plastics which are recommended as referred to earlier in this report. In addition it includes recommendations for the adoption as standard of 14 tentatives and the withdrawal of one tentative. The tentatives and standards in their present form appear in the 1949 Book of A.S.T.M. Standards, Part 6.

NEW TENTATIVE

The committee recommends the following one new tentative for publication as appended hereto.[1]

Tentative Recommended Practice for Injection Molding of Specimens of Thermoplastic Materials:

The committee is recommending this injection molding procedure, considering the wide usage of injection molds today, for obtaining uniform test specimens under like conditions.

REVISIONS OF TENTATIVES

The committee recommends revisions in the following two tentatives:

Tentative Specifications for Melamine-Formaldehyde Molding Compounds (D 704 – 44 T):

The committee recommends that these specifications be revised as appended hereto.[2] These specifications are being revised to include seven instead of four types so that the present range of commercial melamine molding compounds are covered adequately.

Tentative Specifications for Molds for Test Specimens of Plastic Molding Materials (D 647 – 49 T):

An alternate design of injection mold using ejector pins is recommended as an addition to these specifications so that a straight cavity mold can be used to mold test specimens. A further revision also recommended provides a mold for injection molding of test specimens for determination of weld strength. The details of these two new molds are appended to this report.[2]

ADOPTION OF TENTATIVES AS STANDARD

The committee recommends that the following 14 tentatives be approved for reference to letter ballot of the Society for adoption as standard, with revision in one as indicated:

Tentative Specifications for:

Vinyl Chloride-Acetate Resin Plastic Sheets (D 708 – 44 T),
Vinyl Chloride-Acetate Resin Molding Compounds (D 728 – 44 T),
Cast Allyl Plastic Sheet, Rods, Tubes, and Shapes (D 819 – 45 T).

Tentative Methods of Test for:

Luminous Reflectance, Transmittance and Color

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[1] The new tentative specification was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 6.

[2] The revised specifications were accepted by the Society and appear in the 1950 Supplement to Book of ASTM Standards, Part 6.

of Plastic Materials (D 791 - 44 T), revised as appended hereto.[3]
Measuring the Molding Index of Thermosetting Molding Powder (D 731 - 48 T),
Stiffness in Flexure of Plastics (D 747 - 48 T),
Resistance of Plastics to Accelerated Service Conditions (D 756 - 46 T),
Rockwell Hardness of Plastics and Electrical Insulating Materials (D 785 - 48 T), jointly with Committee D-9,
Specific Gravity of Plastics (D 792 - 48 T),
Apparent Density and Bulk Factor of Nonpouring Molding Powders (D 954 - 48 T),
Measuring Shrinkage from Mold Dimensions of Molded Plastics (D 955 - 48 T).

Tentative Recommended Practices for:

Molding Specimens of Amino Plastics (D 956 - 48 T),
Determining Mold Surface Temperature of Commercial Molds for Plastics (D 957 - 48 T), and
Determining Temperatures of Standard A.S.T.M. Molds for Test Specimens of Plastics (D 958 - 48 T).

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends that the following two standards be revised immediately as indicated and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Standard Methods of Test for Index of Refraction of Transparent Organic Plastics (D 542 - 42):

New Section.—Add the following as a new Section 2 on significance of test, renumbering the subsequent sections accordingly:

2. *Significance.*—This test measures a fundamental property of matter useful for control of purity and composition, for simple identification purposes and for optical parts design. It is capable of much greater precision than ordinarily required. The refractometric method is accurate to four significant figures. The microscopic method, which is dependent upon the operator's skill in determining focus, is usually accurate to only three significant figures.

Standard Method of Test for Surface Irregularities of Flat Transparent Plastic Sheets (D 637 - 43):

New Section.—Add the following as a new Section 2 on significance of test, renumbering the subsequent sections accordingly:

2. *Significance.*—This test provides empirical results useful for control purposes and for correlation with service applications. The accuracy is much greater than required for most applications, but it is not expected to measure microscopic defects or the optical perfection of surfaces in terms of wave length of light.

WITHDRAWAL OF TENTATIVE

Tentative Specifications for Nonrigid Vinyl Butyral Plastics (D 745 - 44 T):

The committee recommends that these specifications be withdrawn since only one of the eight grades covered by the specifications is being manufactured commercially, namely, that used in laminated glass. This type is adequately covered by an American Standard (Z26.1–1950).

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[3] The revised method was accepted by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Part 6.

REPORT OF COMMITTEE E-1

ON

METHODS OF TESTING[1]

Committee E-1 on Methods of Testing held two meetings during the year at A.S.T.M. Headquarters in Philadelphia, as follows: on November 17, 1949, and May 16, 1950.

During A.S.T.M. Committee Week in Pittsburgh, Pa., February 27 to March 3, meetings of Subcommittees 1, 2, 4, 6 and 9, and Task Group B were held. Arrangements have also been made for meetings of Subcommittees 2, 5, 6, 9, 14, 15, 21 and 22, and Task Groups A, B, and C during the 1950 Annual Meeting of the Society in June.

The Board of Directors of the Society has appointed Messrs. T. S. Fuller, W. H. Fulweiler, H. F. Moore, and J. R. Townsend each for another term of three years as members of Committee E-1.

A number of changes in subcommittee chairmanships have taken place since last year. J. R. Townsend resigned as chairman of Subcommittee 6 on Indentation Hardness after directing the work of this subcommittee for the past 20 years. R. H. Heyer has been appointed to serve as chairman of this subcommittee. C. E. Barnett resigned as chairman of Subcommittee 11 on Sub-Sieve Testing after having held this position for 12 years. L. T. Work has accepted appointment as temporary chairman of this subcommittee. Robert Burns who served for 12 years as chairman of Subcommittee 14 on Conditioning and Weathering resigned and A. C. Webber has been appointed chairman of this subcommittee. Mr Webber is also chairman of the Joint D-9, D-20 Subcommittee on Conditioning. E. L. Ruh was appointed chairman of the reactivated Subcommittee 21 on Metalware Laboratory Apparatus, replacing W. H. Fulweiler who had accepted the chairmanship temporarily.

The present officers of the committee have been re-elected for the ensuing term of two years.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Method of:

Testing and Standardization of Etched Stem Liquid-in-Glass Thermometers (E 77 - 49 T), and

Compression Testing of Metallic Materials in Sheet Form (E 78 - 49 T).

Revision of Tentative Methods of:

Compression Testing of Metallic Materials (E 9 - 46 T).

Revision of Tentative Specifications for:

A.S.T.M. Thermometers (E 1 - 48 T).

These recommendations were accepted by the Standards Committee on September 22, 1949, and the new and revised tentatives appear in the 1949 Book of A.S.T.M. Standards; E 77 and E 1 in Parts

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[1] **Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.**

3, 4, 5 and 6; and E 78 and E 9 in Parts 1 and 2.

On January 17, 1950, the Administrative Committee on Standards accepted the recommendation of Committee E-1 that the A.S.T.M. Air Chamber Thermometer (A.S.T.M. Reid Vapor Pressure Test) (31 F) be withdrawn from the Standard Specifications for A.S.T.M. Thermometers (E 1 - 49).

NEW TENTATIVES

The committee recommends for publication as tentative the Proposed Method of Verification and Classification of Extensometers, as appended hereto.[2]

The committee recommends that the A.S.T.M. Butadiene Boiling Point Range Thermometer, as appended hereto,[3] be published as tentative and incorporated in the Standard Specifications for A.S.T.M. Thermometers (E 1- 49).

REVISIONS OF TENTATIVES

The committee recommends the following revisions in two tentatives:

Tentative Methods of Verification of Testing Machines (**E 4 - 47 T**):

Section 2(c).—Change this section to read as follows: "(*c*) *Error.*—In the case of a testing machine, the value obtained by subtracting the value indicated by the calibrating device from the value indicated by the testing machine."

Section 8.—In the second sentence change "0.02 per cent" to read "0.1 per cent." In Note 2 change "0.02 per cent" to read "0.1 per cent" in the two places where it appears.

Tentative Methods of Verification of Calibration Devices for Verifying Testing Machines (**E 74 - 47 T**):

Section 2.—Change "0.02 per cent" to read "0.1 per cent" in this section.

Section 16.—Add the following note to this section: "NOTE.—These tests need not be made on a device previously found to comply with the requirements of this paragraph."

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption the following revisions in the Standard Specifications for A.S.T.M. Thermometers (E 1 - 49) and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society.

Section 7.—In the second sentence change "and the manufacturers' name or trade mark" to read "and the manufacturer's or vendor's name or trade mark."

A.S.T.M. Partial Immersion Thermometer (1F - 49).—Change the top of the temperature range from "300 F." to "302 F."; also change the temperature for the dimension from the "Top of the Thermometer to Graduation Line at" from "300 F." to "302 F."

A S.T.M. Stability Test of Soluble Nitrocellulose (26C - 42).—Revise this thermometer, as appended hereto.[3]

ACTIVITIES OF SUBCOMMITTEES AND TASK GROUPS

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman) prepared the new Method of Verification and Classification of Extensometers. Further consideration is being given to other changes in the Methods of Verification of Testing Machines (E 4 - 47 T) and in the Methods of Verification of Calibration Devices for Verifying Testing Machines (E 74 - 47 T), in addition to those recommended in this report.

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[2] This method was accepted as tentative by the Society and appears in the 1950 Supplement to Book of ASTM Standards, Parts 1, 2, and 6.

[3] This recommendation was accepted by the Society; the Standard Specifications appears in their latest form in the 1950 Supplement to Book of ASTM Standards, Parts 3, 4, 5, and 6.

Subcommittee 2 on Effect of Speed in Mechanical Testing (P. G. McVetty, chairman).—A report of the Task Group on the Effect of Speed of Testing on Fatigue Test Results appears as Appendix I. Another Task Group has been studying the speed requirements in A.I.S I., Federal, Navy, and A.S.T.M. specifications in preparation for a proposed revision of Section 20 on speed of testing in the Standard Methods of Tension Testing of Metallic Materials (E 8 - 46). Contemplated changes include a definite distinction between free-running cross head speeds and those measured under load, and the addition of the elasped-time method as another alternative for specifying testing speed. An attempt is being made to stimulate interest in the development of any possible general specification for speed of testing for products for which the A.S.T.M. committees on nonmetallic materials are responsible. No significant progress on this item can be reported at this time.

As a supplement to the 1948 Symposium on Speed of Testing, notes on the speed characteristics of various testing machines and speed effects in tests of a wide variety of materials were compiled and published in the October, 1949, ASTM BULLETIN. Work on the bibliography on speed of testing has been continued by members of the staff of the National Bureau of Standards, and it is expected that the current list issued in 1947 will be brought up to date in 1951.

Subcommittee 3 on Elastic Strength of Materials (F. M. Howell, chairman).—Although the subcommittee has been quite active during the past year, it has no completed action to report other than its recommendation to Committee E-1 of the following statement of its scope: "To implement the functions of Committee E-1 as applied specifically to the initiation, conduct and report of work upon the determination of elastic properties of materials and to prepare methods of determining such properties; to standardize the nomenclature and definitions used in or relating to testing methods and instruments."

There are two task groups actively working on their assignments. The first of these is a joint task group under the chairmanship of P. G. McVetty that is concerned with the correlation of definitions relating to testing methods and instruments. It has made considerable progress in its work of standardizing definitions and is expected to report to the subcommittee at its next meeting. The second task group under the chairmanship of Walter Ramberg is making a survey of the work that has been done in determining the elastic constants of materials and is eventually to recommend suitable methods for the determination of such constants.

Subcommittee 4 on Tension Testing (R. L. Templin, chairman).—During the past year the subcommittee has endeavored to obtain representation from all of the A.S.T.M. committees concerned with the subject of tension testing. This has resulted in an increase in the number of members and has brought to the subcommittee viewpoints which were not previously represented.

Intensive work has been carried out on a revision of the Standard Methods of Tension Testing of Metallic Materials (E 8 - 46) with the idea of incorporating in those methods requirements for the testing of metallic materials not suitably covered at present, so that separate tension testing methods, such as those for steel spring wire (A 318 - 48 T) and for copper rods and bars (B 220 - 48 T), will not be necessary. It is hoped that this revision of Standard Methods E 8 can be completed during the coming year.

Consideration has also been given to the testing of thin metallic materials, although at the present time there are no purchase specifications for such products. A paper on "The Mullen Bursting Strength Test as a Means of Determining the Strength of Annealed Aluminum Foil," was presented by W. C. Aber and F. M. Howell at the Pittsburgh meeting of this subcommittee. The paper appears in Appendix II to this report. A task group has been appointed to study further the tension testing of metallic materials less than 0.005 in. in thickness.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—This subcommittee has undertaken several active programs for revisions of existing standard methods and development of new hardness test methods and hardness conversion tables.

A group under the chairmanship of R. D. France prepared a proposed revision of the Standard Method of Test for Brinell Hardness of Metallic Materials (E 10 – 27).[3a] This revision provides for use of the 1500-kg. load for metals of intermediate hardness, in addition to the 500 and 3000-kg. loads now covered. Another important change covered limits for use of various balls, recommending a steel ball on material having a Brinell hardness number not over 450, a Hultgren ball for not over 500 and a carbide ball not over 630. Suggested loads in the revised methods are based on the following hardness limits: 3000-kg. load 160 to 600 B.H.N., 1500-kg. load between 300 and 80 B.H.N., and 500-kg. load between 100 and 26 B.H.N.

A group headed by V. E. Lysaught has prepared a new hardness conversion table for nickel and high-nickel alloys for consideration by the subcommittee at its June, 1950, meeting.

The subcommittee is sponsoring the technical paper by Frank W. Hussey, "Rockwell C Hardness on Cylindrical Steel Specimens."[4]

Investigation of minimum thickness of materials for Rockwell hardness testing is under way, as part of the activities of a group working on revision of the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 42). Investigations are being made to obtain data for possible revision of the Tentative Hardness Conversion Tables for Steels (E 48 – 43 T).

A tentative method for diamond pyramid hardness tests is being developed and the need for a rapid method of ball indentation hardness testing is under consideration.

Subcommittee 8 on Flexure Testing (H. F. Clemmer, chairman) has completed and is now studying Proposed Methods for Making Three-Edge Bearing Tests of Pipe, which are intended to measure the resistance of pipe to crushing, being particularly applicable to cast iron and concrete pipe, drain tile, etc. Comments received thus far have indicated that some revision is necessary but the subcommittee hopes to be able to present this method to Committee E-1 during the coming year.

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman) has been active through its several task groups as summarized herewith.

The Task Group on Absolute Viscosity Measurements (W. F. Fair, Jr., chairman) has in preparation a pamphlet containing discussions of the various principles by which absolute viscosity may be determined and describing in detail several viscometers that illustrate these principles. Information on the following 21 methods has been compiled and will be considered by the task group at its June, 1950, meeting:

[3a] See Editorial Note, p. 420.

[4] See p. 1176.

	Range, poises
A. Capillary Viscometers:	
1. Bingham Absolute	10^{-2} to 10^{2}
2. Ostwald	10^{-2} to 10^{2}
3. Ubellohde	10^{-2} to 10^{2}
4. Triple Capillary	10^{-1} to 10^{2}
5. Cannon-Fenske	1 to 10^{2}
6. Bingham & Murray	10^{2} to 10^{4}
7. Koppers	1 to 10^{5}
8. Bingham Plastometer	10^{2} to 10^{12}
B. Falling Object Viscometers:	
1. Falling Sphere	10^{2} to 10^{3}
2. Hoeppler	10^{-4} to 10^{4}
3. Falling Cylinder	10^{4} to 10^{8}
C. Rotational Viscometers:	
1. Garrison	10^{-2} to 10
2. Brookfield	10^{-2} to 10^{3}
3. Stormer	10^{-2} to 10^{3}
4. Interchemical	1 to 10^{3}
5. MacMichael	10^{2} to 10^{5}
6. Open End	10^{4} to 10^{8}
7. High Consistency	10^{3} to 10^{9}
D. Parallel Plate Plastometer	10^{7} to 10^{9}
E. Vibrational Viscometers:	
1. Electro-mechanical Transducer	1 to 10^{2}
2. Torsional Crystal Viscometer	1 to 10^{3}

The Task Group on Use of Saybolt Furol Viscosimeter for Asphaltic Materials at High Temperatures (R. R. Thurston, chairman) has been studying a new high-temperature furol viscosity test for asphalts by a cooperative series of tests in seven laboratories on three samples at temperatures of 300, 350, and 400 F. From the results reported to date, it appears that the reproducibility between laboratories is good considering the temperatures and the materials tested. The test results will be reviewed by the task group to determine whether further studies are necessary or if the method is ready for recommendation to Committee E-1 for publication as tentative.

The Task Group on Softening Point (W. A. Kirklin, chairman) is continuing its effort on the task of combining the two A.S.T.M. Ball and Ring Softening Point Tests D 36 – 26 (sponsored by Committee D-4) and E 28 – 42 T (sponsored by Committees D-17 and E-1).

General agreement has been reached on a draft of the combined method. Final recommendations are awaiting the outcome of a current cooperative study by Committee D-4 on the merits and details of stirring the heated bath during the test period.

Of the interested committees, objections have been received from only Committee D-1's Subcommittee XI on Resins which has requested that the word "resin" be removed from the scope of the combined method pending its review of methods for determining softening point of resin. Resin was included in the scope of Method E 28 as a result of earlier studies by Committee D-17.

Subcommittee 11 on Sub-Sieve Testing (C. E. Barnett, chairman) at two meetings held in Chicago, Ill., on June 9, 1949, and June 8, 1950, reviewed the Tentative Recommended Practice for Analysis by Microscopical Methods for Particle Size Distribution of Particulate Substances of Subsieve Sizes (E 20 – 48 T). The subcommittee directed the Task Group on Optical Microscope (R. P. Loveland, chairman) to make a number of changes in this recommended practice.

Three particle size methods under the jurisdiction of other committees have been reviewed. These are Method D 422, Test for Mechanical Analysis of Soils (Committee D-18); Method C 115, Test for the Fineness of Portland Cement by the Turbidimeter (Committee C-1); Method C 204, Test for Fineness of Portland Cement by the Air Permeability Apparatus (Committee C-1). It was concluded that there was no need for action by this subcommittee in respect to these three methods.

The chairman was authorized to appoint a series of temporary small task groups to consider individual methods of particle size measurement and to recommend whether active work should be undertaken on any of them.

Subcommittee 12 on Methods for Density (Wilmer Souder, chairman) considered a set of definitions relating to specific gravity and density of porous solids such as refractories. The definitions had been submitted by the British Ceramic Research Association. A number of comments received were forwarded to the British Association.

Members of the subcommittee have been supplied with a copy of the recent National Bureau of Standards publication "Density of Solids and Liquids," *Circular 487*, March 15, 1950, which will be found helpful on future problems, as it reviews the devices and methods used in this field. The 79 references will be appreciated by those seeking original material.

Subcommittee 14 on Conditioning and Weathering (A. C. Webber, chairman) is continuing its efforts to promote the use by other committees of the established conditions for standard laboratory atmosphere of 23 ± 1.1 C. (73.4 ± 2 F.) at a relative humidity of 50 ± 2 per cent. Several committees have adopted these standard conditions and made suitable changes last year in the standards under their jurisdiction. Other committees have the matter under consideration.

The subcommittee plans to sponsor a Symposium on Conditioning and Weathering in order to bring together the vast amount of information and experience that has accumulated in the past several years.

Subcommittee 17 on Thermometers (R. M. Wilhelm, chairman) completed a number of editorial changes in the Thermometer Specifications E 1 and also developed the Method of Testing and Standardizing Thermometers (E 77 – 49 T). It is presenting specifications for one revised standard thermometer and one new tentative thermometer as mentioned earlier in this report. Specifications for a set of 18 precision fractionally graduated test thermometers have been completed and will be presented to the Society through the Administrative Committee on Standards subsequent to the Annual Meeting.[3a] Consideration is being given to a mercury thallium thermometer for low cloud and pour test and also to specifications for a thermometer for furol viscosity test at high temperatures. Further study is also being made of the new methods of testing thermometers and it is expected that some changes will be recommended during the year.

Subcommittee 18 on Hydrometers (J. P. Bader, chairman) has reactivated and reorganized its work. The personnel of the subcommittee is being enlarged to include representatives of manufacturers as special adviser members and representatives from other technical committees interested in the use of hydrometers. The subcommittee has completed reviews of three Standard Methods of Test for: Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (D 287 – 39), for Mechanical Analysis of Soils (D 422 – 39), and for Specific Gravity of Creosote (D 368 – 33). A number of suggestions for improvement of the hydrometers used in these tests have been submitted to Committees D-2, D-18 and D-7, responsible respectively for preparation of these methods.

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman) is cooperating with Committee D-2 on Petroleum Products and Lubricants on a project to consider the establishment of methods for calibrating cylindrical and pear-shaped centrifuge tubes. On the recommendation of this subcommittee, Committee E-1 plans to appoint a task group to standardize the nomenclature of porous filtering bodies and methods of test for determining the porosity of these filters. Considerable informal assistance has been rendered other technical committees in the preparation of specifications for laboratory glassware.

Subcommittee 21 on Metalware Laboratory Apparatus (E. L. Ruh, chairman) has been reactivated and reorganized under its new chairman. A number of manufacturers of this type of apparatus have been added to the committee as special adviser members and additional representation is being obtained from other A.S.T.M. technical committees. At its June 1950, meeting, plans will be made for handling promptly and efficiently all of the matters that rightly come under the surveillance of this subcommittee. Consideration will be given to problems recently brought to the attention of the subcommittee. It will also consider the results of a survey being made of manufacturers and distributors of laboratory apparatus and instruments through the cooperation of the Scientific Apparatus Makers of America. In addition, the members of the subcommittee are being asked, through a questionnaire, to propose any matters for attention of the subcommittee.

Task Group A on Distillation Tests (H. P. Ferguson, chairman) held its organization meeting at Atlantic City on June 28, 1949, and discussed plans for rationalizing the differences between the present A.S.T M. Distillation Methods D 86, D 268, D 850, D 1078, and D 216. At the initial meeting, the group was receptive to the idea of simplifying the test methods but emphasis was placed on the commercial implications of changes made without serious study.

A survey of the various A.S.T.M. thermometers used in distillation tests will be reviewed by the task group at its next meeting. Progress in simplification here will aid the committee in its goal to correlate and simplify the present methods.

Task Group B on Low-Temperature Testing of Elastomers and Plastics (R. S. Havenhill, chairman).—This task group was organized in June, 1949, in view of the urgent need for standardization and correlation of research in the field of low-temperature testing of elastomers and plastic materials. Its scope includes an analysis of the testing methods now being used, both A.S.T.M. and others, in various research programs under way in industry, government agencies, and educational institutions. This analysis will include the extent as well as the suitability of present tests and will cover both the standardization of present methods and the development of new standard methods. A questionnaire has been sent out to the various industries, government agencies, educational institutions, and research centers to determine what low-temperature tests are being used and whether present A.S.T M. tests are adequate.

Task Group C on Water Vapor Permeability (R. Burns, chairman) at its organization meeting in June, 1949, appointed a section consisting of representatives of Committees C-16 on Thermal Insulating Materials, D-6 on Paper and Paper Products, and D-20 on Plastics to review the existing methods for water vapor permeability prepared by these committees. A proposed method prepared by the section is now under review in the task group. It is applicable to the testing of papers, textiles, plastics, all kinds of building papers (roofing paper), building board, and plaster board.

This report has been submitted to letter ballot of the committee, which consists of 12 members; 12 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. R. TOWNSEND,
Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:
ASTM Thermometers (E 1 - 50).

Revision and Reversion to Tentative of Standard Method of:
Test for Brinell Hardness of Metallic Materials (E 10 - 27).

These recommendations were accepted by the Standards Committee on September 26, 1950. Tentative Specifications E 10 appear in Parts 1 and 2 of the 1950 Supplement to Book of ASTM Standards, and the tentative revision of E 1 appears in Parts 3, 4, 5, and 6 of the Supplement.

APPENDIX I

REPORT OF THE TASK GROUP ON EFFECT OF SPEED OF TESTING ON FATIGUE TEST RESULTS

At temperatures and stresses below those which cause continuing flow under a steady load, the effect of speed of testing, that is the effect of frequency of application of stress cycles to a specimen or a structural part, is relatively small. Above the "creep temperature," the effect of frequency of cycles is, in general, much larger. This report, then, is divided into two sections.

EFFECT OF SPEED OF TESTING ON FATIGUE TEST RESULTS FOR METALS TESTED BELOW THE "CREEP" RANGE OF TEMPERATURE

A stress is not set up simultaneously all through a structural member but spreads from an exciting source through the member at the rate of $v = \sqrt{Eg/w}$, where E is the modulus of elasticity, w is the weight per cubic inch of the metal, and g is the acceleration due to gravity. For steel this means that the stress travels through the metal at the rate of 17,200 ft. per sec. However, it is hardly to be expected that variations in the frequency of stress cycles would materially affect the endurance properties unless considerable heat is produced by damping of vibrations with resulting mechanical hysteresis. Such heating does occur, especially on large pieces, and in some cases it has been found necessary to test large rotating-beam specimens at a slow speed, or to use elaborate methods of cooling.

Some further study of hysteresis, damping properties of metal, and speed of making fatigue tests seems desirable.

Unless they are under simultaneous cyclic stressing and corrosion, small metal specimens which run below the "creep temperature" are not very seriously affected by varying frequency of stress cycles—at least between the limits of 200 to 7000 stress cycles per minute (**1, 2, 3, 4**).[1]

Under conditions involving simultaneous stress cycles and corrosion, the spread of damage seems to depend on *time* as well as on number of cycles of stress (**5**). Hence in slow-speed tests, specimens fail under fewer cycles of a given stress than is the case in high-speed tests.

The dependence of damage by corrosion on time as well as on number of stress cycles may explain why under corrosion-fatigue there seems to be no endurance limit for an indefinitely large number of stress cycles. The S–N diagram is a *downward* sloping line even after hundreds of millions of stress cycles. No matter how long a corrosion-fatigue test continues, the corrosive liquid will form a thin skin of corroded metal, and the repeated stress cycles may crack that skin, exposing fresh metal to the corroding liquid, and a spreading crack seems sure to lead "at long last" to fracture.

EFFECT OF SPEED OF TESTING ON FATIGUE TEST RESULTS FOR METALS TESTED AT TEMPERATURES UNDER WHICH CONTINUING FLOW OR "CREEP" OCCURS

Creep of metals is dependent on *time*, while "fatigue" of metals is commonly

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[1] The boldface numbers in parenthesis refer to the list of references appended to this report, see p. 424.

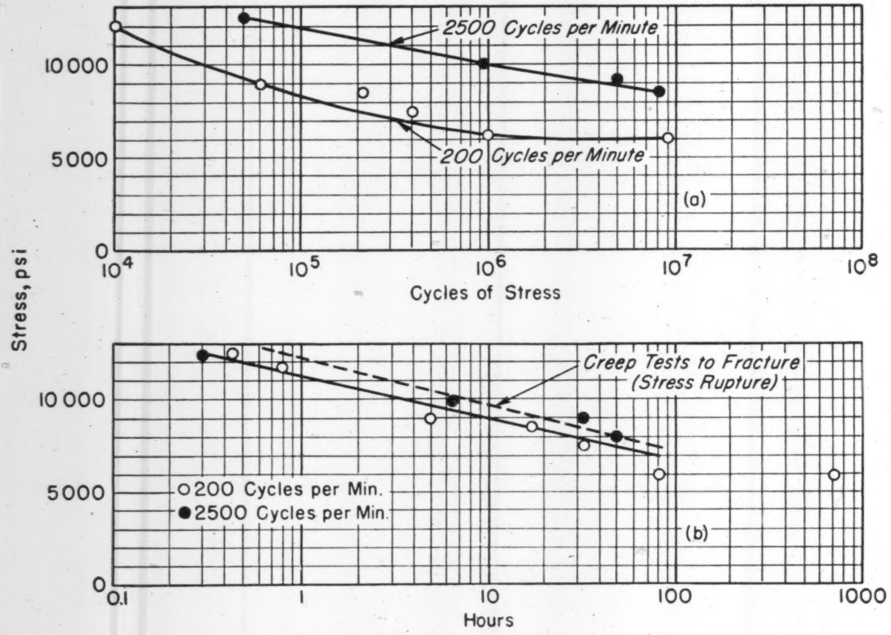

FIG. 1.—Reversed-Flexure Fatigue Tests of Structural Steel at 1200 F.

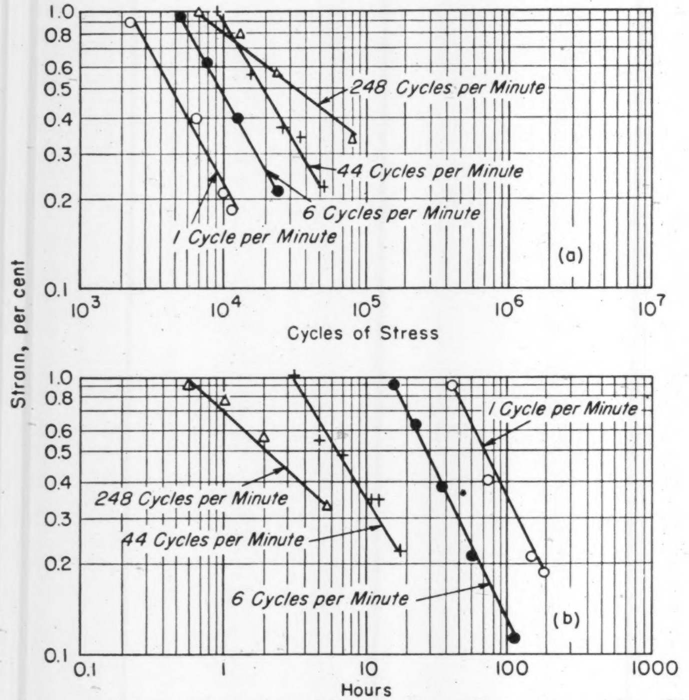

FIG. 2.—Reversed-Flexure Tests of Lead Sheet at Room Temperature (about 74 F.).

regarded as a function of number of stress cycles. Either creep or cycles of stress may cause a progressive fracture with greatly reduced ductility. Creep produces its fracture by a load continued for a length of time, while repeated stress cycles develop a spreading damage as some function of the *number of cycles* of stress applied. A structural part (or a specimen) may be subjected to creep conditions without repeated stress, but, above the minimum temperature to produce appreciable creep, the part cannot be subjected to repeated stress without the occurrence of creep, and at length the fracture of the piece. This combined action of repeated stress cycles and of creep is somewhat similar in its destructive effects to those of combined repeated stress and corrosion noted above.

In the fatigue tests of steel at high temperature, reported by Toolin and Mochel (6), the *S–N* diagrams do not become horizontal but are downward sloping lines even after 100,000,000 cycles of stress.

If stress cycles are applied only a few times a minute, then for a given number of cycles the *time* will be longer and more structural damage will be done by *creep* than if stress cycles were applied at a higher frequency. This is illustrated in Figs. 1 and 2. In Fig. 1 (*a*), an ordinary *S–N* diagram is shown for structural steel with a carbon content of 0.17 per cent, the test being conducted at a temperature of 1200 F.,—a temperature under which structural steel shows a quite high creep rate. One set of specimens was tested with a stress cycle frequency of 200 per minute, and the other set was tested with a stress cycle frequency of 2500 per minute. It will be noted that the *S–N* diagram for the 2500 cycle per minute tests lies distinctly *above* the *S–N* diagram for the 200 cycle per minute tests.

In Fig. 1 (*b*), maximum stress is plotted as ordinates and *time* elapsed during the test of a specimen as abscissas. For this particular metal at 1200 F., *one* stress-time diagram fits both the tests at 200 stress cycles per minute and the tests run at 2500 cycles per minute. This coincidence is probably not significant, although it would seem to indicate that in these particular tests creep was the dominant factor causing progressive fracture. In Fig. 1 (*b*), there is also shown a "stress-rupture diagram" for this metal, that is, a curve plotted with values of steady tensile stress as ordinates and of time for fracture as abscissas. This "stress-rupture diagram" is slightly "higher" than the stress-time diagram for the fatigue tests of the metal. Figure 2 shows results of tests of lead[2] strips tested in reverse flexure at room temperature (about 74 F). It will be noted in Fig. 2 (*a*) that the lower the frequency of stress cycles the fewer the number of stress cycles were required for fracture under a given maximum strain. For a reversed strain of ±0.4 per cent applied at the rate of one cycle per minute, the number of cycles for fracture is only about one-quarter the number required if ±0.4 per cent strain is applied at the rate of 44 cycles per minute, and only about one-tenth the number required for 248 cycles per minute.

However, if the test results for the lead strip tests are plotted on a strain-time diagram, as is done in Fig. 2 (*b*), it is seen that for a given magnitude of reversed cycle strain the fewer the stress cycles per minute, the *longer* the *time* required for fracture.

The data available today are not nearly sufficient to warrant any quantitative statement of effects of combined creep and repeated stress or combined repeated stress and simultaneous corrosive action. However, it is evident that in reporting fatigue test results *the fre-*

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[2] Lead shows measurable creep at temperatures as ow as 32 F.

quency of application of stress cycles should always be reported.

SUGGESTED LINES OF FURTHER RESEARCH

The following are suggested for future lines of research:

1. Tests using various frequencies of stress cycle and various stress ranges during the cycle. Most test data available are for cycles of completely reversed stress.

2. Tests of metals like aluminum and magnesium which show but a minute amount of creep at room temperature, but whose *S–N* diagrams frequently do not show an endurance limit for indefinitely large numbers of stress cycles,—that is, the *S–N* diagram continues to be a downward sloping line even after (say) 500,000,000 cycles of stress.

3. Tests of non-ferrous metals, plastics, and possibly ceramic materials and textiles.

4. Tests to study the relation of damping capacity of metals to the results of creep and fatigue tests at elevated temperatures.

Respectfully submitted,

GEORGE R. GOHN,
F. M. HOWELL,
BRUCE L. WILSON,
H. F. MOORE, *Chairman.*

REFERENCES

(1) T. T. Oberg and J. B. Johnson, "Fatigue Properties of Metals Used in Aircraft Construction at 3450 and 10,600 Cycles," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 195 (1937).

(2) Test Data from Talbot Laboratory, University of Illinois.

(3) G. N. Krouse, "A High-Speed Fatigue Testing Machine and Some Tests of Speed Effect on Endurance Limit," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 156 (1934).

(4) H. F. Moore and G. N. Krouse, "Repeated Stress (Fatigue) Testing Machines Used in the Testing Laboratory of the University of Illinois," *Circular 23*, University of Illinois Engineering Experiment Station (1934). See *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 506 (1939).

(5) R. J. McKay and R. Worthington, "Corrosion Resistance of Metals and Alloys," McGraw-Hill Publishing Co., Inc., New York, N. Y. (1936).

(6) P. R. Toolin and N. L. Mochel, "The High-Temperature Fatigue Strength of Several Gas Turbine Alloys," *Proceedings*, Am. Soc Testing Mats., Vol. 47, p. 677 (1947).

APPENDIX II

THE MULLEN BURSTING STRENGTH TEST AS A MEANS OF DETERMINING THE STRENGTH OF ANNEALED ALUMINUM FOIL

By W. C. Aber[1] and F. M. Howell[1]

The present Standard Methods of Tension Testing of Metallic Materials (E 8 - 46)[2] limit the use of the standard sheet tension test specimen to material not less than 0.005 in. in nominal thickness. One reason for this is that it is appreciated that the testing of thinner material introduces some difficulties that either do not exist in the testing of thicker material or, if they do exist, they do not have an important effect upon the test results. Another reason is that, at the time the methods were formulated, there was no need for a method of testing material thinner than 0.005 in. Recently, however, there have been some indications that there should be some means of evaluating the strength of thin metallic materials to assure a purchaser that he is getting material that has the same characteristics as material he has found satisfactory.

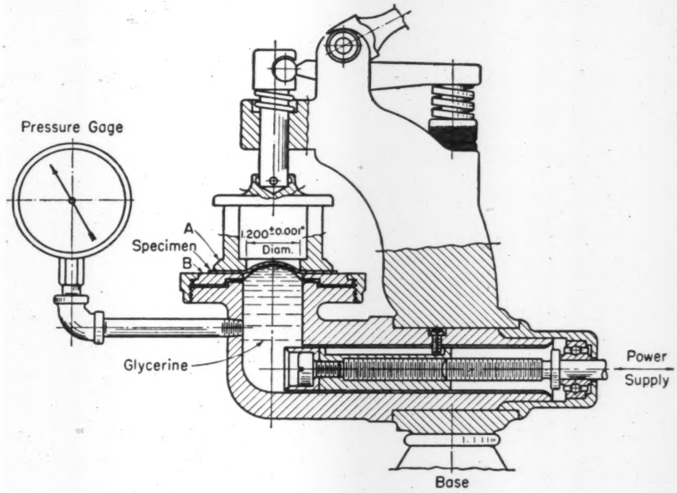

Fig. 1.—Schematic Diagram Illustrating Apparatus for Making Mullen Bursting Tests.

In the tension testing of thin materials, the most difficult problem is to prepare suitable specimens. Generally they cannot be milled or shaped satisfactorily but must be cut to size and shape by means of a shear. Such a shear must produce specimens that are free from nicks, burrs, or discontinuities of any kind. Furthermore, there is another important problem, that of applying load uniformly across the width of the specimen so that it will fail in pure tension rather than by a tearing action.

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[1] Research Labs., Aluminum Company of America, New Kensington, Pa.

[2] 1949 Book of A.S.T.M. Standards, Part 2, p. 993.

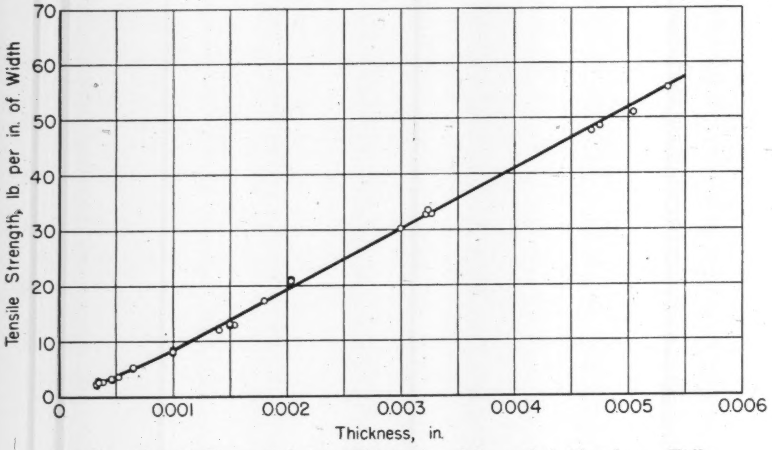

FIG. 2.—Tensile Strength *versus* Thickness of Annealed Aluminum Foil.

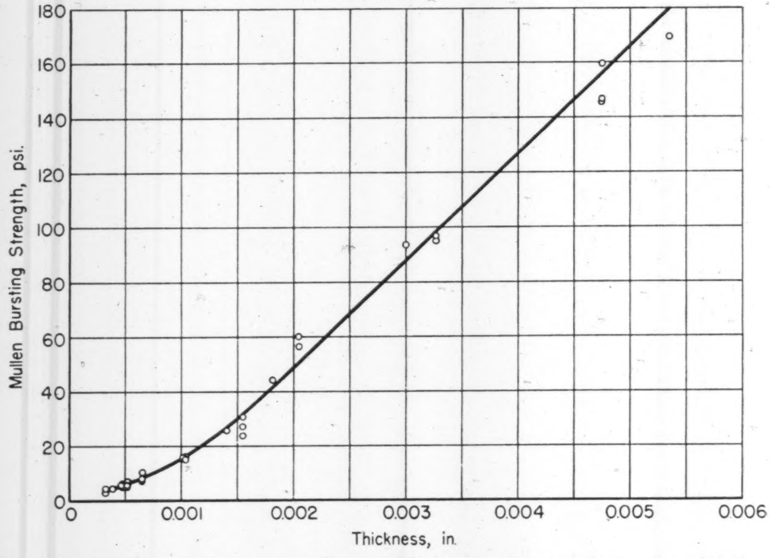

FIG. 3.—Mullen Bursting Strength *versus* Thickness of Annealed Aluminum Foil.

For many years we have made tension tests of annealed aluminum foil to obtain data on its strength characteristics. The most suitable method is one that complies essentially with the requirements of A.S.T.M. Standard Method of Test for Tensile Breaking Strength of Paper and Paper Products (D 828 - 48).[3] The speci-

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[3] *Ibid*, Part 4, p. 989.

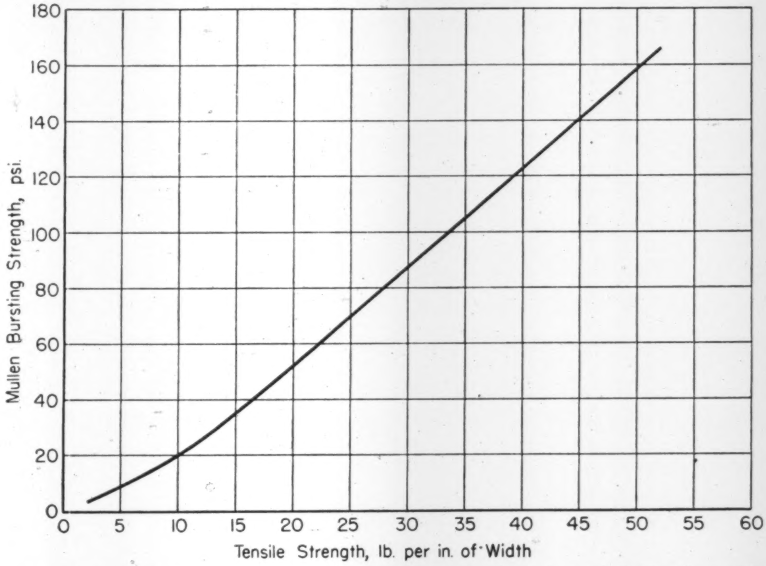

FIG. 4.—Mullen Bursting Strength *versus* Tensile Strength of Annealed Aluminum Foil.

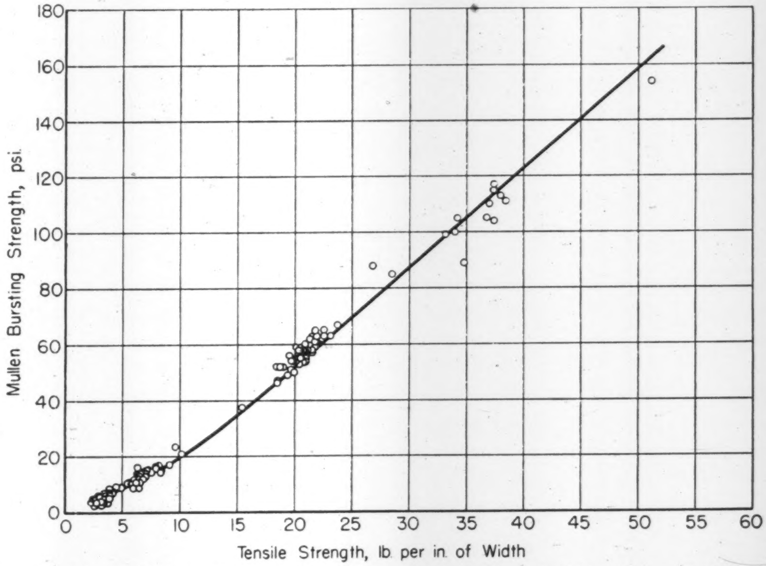

FIG. 5.—Comparison of Plant Data on Production Lots of Annealed Aluminum Foil With Mullen Bursting Strength - Tensile Strength Curve.

mens used are $\frac{1}{2}$ in. wide by at least 10 in. long, and they must have smooth, clean edges to give satisfactory results. Even small nicks and burrs that are too small to be seen by the unaided eye or cold working of the edges during shearing have appreciable effects on the tensile strength and elongation values obtained.

Because of the difficulties involved in obtaining satisfactory results from tension tests, other kinds of tests have been investigated in an attempt to find one that is not so critically affected by test specimen details. The most satisfactory test seems to be another of the tests that has for a long time been used in testing paper. This is the A.S.T.M. Standard Method of Test for Bursting Strength of Paper (D 774 – 46),[4] commonly known as the Mullen test.

A schematic diagram illustrating apparatus for making Mullen bursting tests is shown in Fig. 1. The specimen is clamped between two circular plates, *A* and *B*, containing circular openings. Beneath the lower plate *B* is a pure gum rubber diaphragm which is the top cover of a reservoir completely filled with glycerine. In making tests, pressure is applied to the glycerine, usually by means of a motor-driven piston, which deforms the rubber diaphragm and applies pressure to the specimen which ruptures as soon as its bursting strength has been exceeded. The pressure exerted by the glycerine at the time of bursting is indicated by a hydraulic pressure gage connected to the reservoir containing the glycerine. The Mullen bursting strength value is the reading of the pressure gage at the time of failure of the specimen.

One of the definite advantages of a test of this kind is that there is no edge preparation of the specimen involved, since it is only the central portion of a relatively large area of the specimen that is deformed during the test. It has also been found that in the case of annealed aluminum foil the results of such tests can be correlated quite satisfactorily with the results of the much more costly tensile strength tests.

To define the relation between bursting strength values and tensile strength values, 17 lots of annealed aluminum foil ranging in thickness from 0.00035 to 0.005 in. were carefully tested by both methods. The tensile strength values plotted in Fig. 2 show the relation between thickness and tensile strength in pounds per inch of width. It will be noted that throughout the thickness range of 0.001 to 0.005 in. the increase in strength is proportional to the increase in thickness, but for thinner material the strength is less than is indicated by the straight-line relationship. A similar plot of Mullen bursting strength values against thickness is shown in Fig. 3. Here again there is a straight-line relationship between Mullen bursting strength and thickness from a thickness of about 0.0015 in. up to a thickness of 0.005 in. For thinner material, the bursting strength is proportionately less.

From these two sets of data, the curve of Fig. 4 was drawn to show the relation between Mullen bursting strength and tensile strength. This curve is a straight line for bursting strengths greater than 40 psi. This same curve is presented in Fig. 5 with the results of tension and bursting tests of some 200 lots of foil taken from commercial production over a period of about 15 months. They agree quite well with the curve, thus supporting the previously established relationship.

Referring back to Figs. 2 and 3, it will be observed that the scatter of results is greater for the Mullen bursting strength values than for the tensile strength values. It should be appreciated, however, that the tension tests were very carefully made by a man who has had almost 20 years of experience with work of this kind and who is quite expert in detecting and discarding specimens with nicks, burrs, and other irregularities that would affect the results. No such care and experience is required to make bursting tests. If the machine is maintained in good condition, even an inexperienced technician can obtain good results. Even though there may be somewhat more scatter in results than is shown by very carefully conducted tension tests, the scatter is much less than might be encountered in tension tests when all precautions are not observed in preparing and making the tension tests.

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[4] *Ibid*, Part 4, p. 956.

To make a tension test requires a shear for preparing specimens and a testing machine for testing them. To make a bursting test requires only a Mullen tester. The time required to make a bursting test is only about one-fourth that required to make a tension test. The cost, therefore, both as to investment in equipment and man-hours to conduct the test is very much in favor of the bursting test.

It is believed that the relatively simple Mullen bursting strength test provides an adequate measure of the strength of annealed aluminum foil, and it is recommended that for those demanding a specification requirement of strength, such requirement be based on the bursting strength rather than the tensile strength.

DISCUSSION

MR. M. E. MARKS.[1]—A.S.T.M. Committee D-20 on Plastics has been cooperating with the Society of the Plastics Industry in developing a test for determining the strength of thin plastic films. At present it is believed that a test such as the Mullen bursting strength test may be useful. Would Mr. Howell care to comment as to the possibility of using his procedure for testing such films of plastic?

MR. F. M. HOWELL (*author*).—I am not in a position to comment on the usefulness of the Mullen test for very ductile thin plastic films, as we have not made any Mullen tests on such materials. As you know, this test has been used for years by the paper industry and has been found very useful in that field.

MR. H. E. SMITH.[2]—One thing that perhaps may be worth while keeping in mind is that many Mullen testers are operated by hand, and I have found in testing heavy paper that it is hardly possible to operate them uniformly enough to be sure of a fair application of the load.

I would think that with thin metals, foils, etc., the same might apply, so it is desirable to operate the machine by a small electric motor.

MR. HOWELL.—We find that the motor-driven tester has several advantages, one of them being that the pressure is built up, if not at a uniform rate, at least in a reproducible manner. In testing aluminum we are fortunate that we have a material that is not particularly sensitive to the speed effect. Nevertheless, we prefer a motor-driven tester.

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[1] Research Chemist and Supervisor of Organic Research Testing Lab., Columbia Chemical Div., Pittsburgh Plate Glass Co., Barberton, Ohio.

[2] Materials Engineer, White Plains, N. Y.

REPORT OF COMMITTEE E-2
ON
EMISSION SPECTROSCOPY[1]

Committee E-2 on Emission Spectroscopy held two meetings during the year, the first at Atlantic City, N. J., on June 28, 1949, and the second in Pittsburgh, Pa., on February 16, 1950. Meetings of the Advisory Committee also were held in Atlantic City, N. J., on June 27, 1949, in Buffalo, N. Y., on October 28, 1949, and in Pittsburgh, Pa., on February 16, 1950. The membership of the committee is now 117, representing a net gain of 8 members over last year.

The committee recommends no change in status of its present tentative methods.

Committee E-2 activities were carried out through the subcommittees, all of which were active. Considerable progress has been made in writing and reviewing suggested methods of analysis, which now total 48, which it is proposed to publish as an E-2 Book of Methods. In addition, the subcommittees considering the more fundamental aspects of spectrographic analysis have been organized into task groups that have completed several assignments and made substantial progress in others. An account of these activities during the year are given for each subcommittee.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee E-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Withdrawal of Tentative Methods of:

Quantitative Spectrochemical Analysis of High Grade Pig Lead (E 25 – 37 T), and

Spectrochemical Analysis of Lead Alloys for Minor Constituents and Impurities (E 49 – 43 T).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (R. W. Callon, chairman) has prepared a report on cooperative testing of a new S. A. No. I type photographic emulsion prepared by Eastman Kodak Co. The consensus of the group testing the emulsion was that it was more uniform in contrast as a function of wavelength but that its reduced sensitivity was a marked disadvantage. The subcommittee, through a group headed by R. G. Russell, has been engaged in the writing of specifications and tolerances for spectrographic equipment. A preliminary report has been prepared and circulated within the subcommittee. The third activity of the subcommittee has been the revision of a report by G. A. Nahstoll on Location, Installation, and Safety Precautions for the Spectrographic Laboratory. This report is in final form for publication in the forthcoming Committee E-2 book of methods. The subcommittee plans further action on specifications for equipment and on the testing of experimental photographic emulsions.

Subcommittee II on Fundamental Meth-

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

ods (R. W. Smith, chairman) has been organized into seven groups which have undertaken the preparation of suggested practices in basic procedures as follows: (*1*) R. A. Wolfe, C. H. Corliss, and G. H. Dieke prepared a report on the Specification of Spark Generators which is ready for publication in the E-2 book of methods; (*2*) J. H. Coulliette prepared a report on Calculations in the Mutual Standard Method of Spectrochemical Analysis which is to be distributed to the subcommittee; (*3*) J. Sherman has prepared a 40-page recommendation for Evaluation of Precision and Accuracy in Spectrochemical Analysis; (*4*) C. Feldman has undertaken the preparation of a report on Photometric Practices which is expected to be finished in about six months; (*5*) D. Fry has presented a preliminary report on Rapid Semi-Quantitative Spectrochemical Analysis but further work remains to be done in evaluating the methods; (*6*) M. F. Hasler is preparing a report on the Internal Standard Method of Spectrochemical Analysis; and (*7*) H. A. Tuttle is preparing a Suggested Method for Photographic Processing which will be included in the E-2 book of methods.

Subcommittee III, Editorial (C. L. Guettel, chairman) has been engaged principally in reviewing and editing the 48 analytical methods prepared by Subcommittees V through X. Of these methods, 36 have been released by Subcommittee III and distributed to Committee E-2. It is expected that all of the methods will be ready for publication by the end of the year. A report on The Available Translations and Translating Services for Spectrochemical Analysis has been prepared by W. M. Henry and is to be edited for publication.

Subcommittee IV on Standards and Pure Materials (C. H. Corliss, chairman) has been engaged in the revision of its Report on Standard Samples for Spectrochemical Analysis. This report which is published every three years by the A.S.T.M. will be released for publication within two to three months.[2] The report will include an expanded section on pure materials.

Subcommittee V on Copper, Nickel, and Their Alloys (E. K. Jaycox, chairman) has prepared nine analytical methods covering the analysis of copper, nickel, bronze and admiralty metal. Four of these have been edited and distributed to Committee E-2.

Subcommittee VI on Lead, Tin, Antimony, Bismuth and Their Alloys (E. J. Dunn, Jr., chairman) has prepared nine methods, of which six have been distributed to Committee E-2. The methods cover tin and lead and various tin-lead alloys, including solder.

Subcommittee VII on Aluminum, Magnesium and Their Alloys (J. R. Churchill, chairman) has prepared and edited three methods, of which two are for aluminum alloys and one is for magnesium alloys.

Subcommittee VIII on Zinc, Cadmium and Their Alloys (A. Goldblatt, chairman) has prepared six methods which have been distributed to Committee E-2. These include zinc, cadmium, and zinc die-casting alloys.

Subcommittee IX on Ferrous Metals (P. R. Irish, chairman) has prepared ten methods covering the analysis of cast iron and low-alloy steel. The methods have been distributed to Committee E-2.

Subcommittee X on Non-Metals (R. H. Bell, chairman) has prepared ten methods which include the analysis of silica refractories, ceramics, alkaline earth titanates, lithium-bearing ores, soda ash, silver nitrate, caustic soda, strike solutions (for silver), and bright-nickel plating solutions (for Zn).

The 48 suggested analytical methods

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[2] Issued as a separate publication, *STP No. 58-A*, October, 1950.

and four related reports, including that of the Joint-Committee on Nomenclature in Applied Spectroscopy, are expected to be submitted for publication to A.S.T.M within a few months.

This report has been submitted to letter ballot of the committee, which consists of 117 members; 76 members returned their ballots of whom 68 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

B. F. SCRIBNER,
Chairman.

MARY E. WARGA,
Secretary.

REPORT OF COMMITTEE E-3

ON

CHEMICAL ANALYSIS OF METALS[1]

A report of Committee E-3 would not be complete without an acknowledgment of the loyalty and hard work evidenced by a large number of individuals who make up its membership. Any recital of statistics cannot do justice to the careful thought and critical review which have gone into the methods which finally appear under the sponsorship of this committee. These methods, even after they have been issued, are under constant scrutiny to keep them up to date and to eliminate weak points. The new edition of the Book of A.S.T.M. Methods of Chemical Analysis of Metals, which is now in preparation, will give evidence of the results of these efforts.

A meeting of Committee E-3 was held at the Annual Meeting of the Society on June 29, 1949, which served to correlate the many activities of its divisions and subcommittees.

The Advisory Committee, which serves as the clearing house for establishing the broad policies of Committee E-3 between meetings, has held two meetings during the year: on June 26, 1949, at Atlantic City, N. J., and on February 16, 1950, in Pittsburgh, Pa.

The Advisory Committee has been concerned because a considerable number of members of Committee E-3 are not active in the work of the committee and efforts will be made to stimulate greater activity.

The Advisory Committee approved the recommendation that the composition range for an element to be determined as given in the scope of E-3 methods should be limited by the smallest amount, consistent with manipulative technique, that can be determined by the method and the largest amount that can be found in the material and determined by the method.

The Editorial Subcommittee (S. E. Q. Ashley, chairman) has been very active in reviewing broad editorial policies for Committee E-3 and in setting standards to be followed in the preparation, review, and final editing of analytical procedures. Tentative outlines to be followed in writing new methods and a routing plan to be followed in the review of methods have been circulated to the members of Committee E-3. Four meetings of the subcommittee have been held: at the International Testing Laboratories, Inc., Newark, N. J., on September 30, 1949; at the Chemists Club, New York, on December 16, 1949, and January 20, 1950; and at Society Headquarters, Philadelphia, on April 12, 1950. Two members from each of Divisions F and N have been added to the editorial subcommittee to provide direct representation from these Divisions and to assist in the editorial review of methods originating within the Division. The following recommendations on broad editorial policies have been approved:

Data obtained in analytical procedures should be "rounded off" to the desired number of places in accordance with the rounding off method of the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (E 29 - 49 T),[2] Section 3 (*d*) through (*h*).

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

The final editorial review of a new method shall be made by the chairman of the division in which the method originates with the assistance of his representatives on the editorial subcommittee.

A special task group has been set up by the editorial subcommittee to consider a reorganization of the arrangement of methods in the Book of A.S.T.M. Methods for Chemical Analysis of Metals.

Nine new members were added to the committee during the year and two members resigned. The active membership of the committee is now 102.

The election of officers for the ensuing two years resulted in the following:

Honorary Chairman, H. A. Bright.

Honorary Vice-Chairman, C. B. Francis.

Chairman, J. W. Stillman.

Vice-Chairman, Arba Thomas.

Secretary, H. Kirtchik.

Members of the Advisory Committee At Large, J. L. Hague, Calvin Sterling.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative:

Photometric Method for Determination of Cobalt in Nickel (E 39 - 49 T), and

Photometric Methods for Chemical Analysis of Aluminum and Aluminum-Base Alloys (covering the determination of chromium, copper, iron, manganese, nickel, and titanium) (E 34 - 49 T).

These recommendations were accepted by the Standards Committee on September 22, 1949, and the new tentative methods will appear in the 1950 Book of A.S.T.M. Methods on Chemical Analysis of Metals.

NEW TENTATIVES

The following new methods, when approved by Committee E-3, will be submitted to the Administrative Committee on Standards for acceptance as tentative.

Methods for the Chemical Analysis of Nickel-Copper Alloys,

Methods of Chemical Analysis for Sulfur in Special Brasses.—These methods will be, in effect, a tentative revision of the Standard Methods of Chemical Analysis of Special Brasses and Bronzes (E 54 - 49),[3]

Methods for the Chemical Analysis of Antimony,

Photometric Methods for the Chemical Analysis of Lead, Tin, Antimony, and Their Alloys, and

Tentative Method of Chemical Analysis for Aluminum in Zinc-Base Die-Casting Alloys.—This method will be, in effect, a tentative revision of the Standard Methods of Chemical Analysis of Zinc-Base, Die-Casting Alloys (E 47 - 45).[3]

REVISION OF TENTATIVES

Revisions in the following tentatives, when approved by Committee E-3, will be submitted to the Administrative Committee on Standards.

Tentative Recommended Practice for Photometric Methods for Chemical Analysis of Metals **(E 60 - 46 T)**.[3]

Tentative Photometric Method for the Determination of Boron in Steel **(E 30 - 48 T)**.[4]—The revision consists of the addition of new methods for determining beryllium and tin in steel.

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[2] 1949 Book of A.S.T.M. Standards, All Parts.

[3] 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals.

[4] Issued as a separate reprint.

Photometric Methods of Analysis of Aluminum Alloys (**E 34 - 49 T**).[4]—The revision of these methods consists of the addition of a procedure for the determination of bismuth and several changes in the procedures already included in the methods.

Photometric Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (**E 61 - 46 T**).[3]—The revision consists of the addition of new methods for determining copper and nickel in magnesium and magnesium-base alloys.

Tentative Methods of Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys (**E 75 - 49 T**).[4]—The committee recommends the incorporation of the salicylate method for iron, formerly published under the designation E 63 - 46 T.[3]

Tentative Photometric Methods for Chemical Analysis of Copper and Copper-Base Alloys (**E 62 - 46 T**).[3]—The revision consists of the addition of a new method for determining silicon in copper-base alloys.

Tentative Photometric Methods for Determination of Iron in Slab Zinc (Spelter) (**E 64 - 46 T**).[3]—The committee recommends the deletion of the ferric chloride and salicylate methods from this tentative and the retention, without change, of the thiocyanate method.

REVISION OF STANDARD, IMMEDIATE ADOPTION

Committee E-3 recommends for immediate adoption the revision of the following recommended practice and accordingly asks for a nine-tenths affirmative vote at the Annual Meeting in order that these recommendations may be referred to letter ballot of the Society:

Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (**E 50 - 48**).[3]—The revision, which is appended,[5] consists primarily of a revised table of concentrations of acids and ammonium hydroxide, a revised section on hazards, and revisions in requirements for a number of reagents. There will also be added to Recommended Practice E 50, as an editorial revision (not appended), a table listing all the reagents used in the E-3 methods.

WITHDRAWAL OF STANDARD

It is recommended that the *Standard Method of Sampling Slab Zinc (Spelter)* (**E 65 - 48**)[3] be withdrawn since the procedure has been incorporated in the Standard Specifications for Slab Zinc (Spelter) (B 6 - 49),[6] which are under the jurisdiction of the A.S.T.M. Committee B-2 on Non-Ferrous Metals and Alloys.

WITHDRAWAL OF TENTATIVES

It is recommended that the following tentatives be withdrawn:

Photometric Method for Determination of Bismuth in Pig Lead (**E 58 - 45 T**).[3]—This procedure is being incorporated in a revision of Methods of Chemical Analysis of Pig Lead (E 37 - 45)[3] which will be submitted to the Administrative Committee on Standards for publication as tentative.

Photometric Method for Determination of Iron in 70–30 Copper-Nickel Alloy (**E 63 - 46 T**).[3]—A revision of the Tentative Methods of Chemical Analysis of Copper-Nickel and Copper -Nickel-Zinc Alloys (E 75 - 49 T)[4] to incorporate this procedure will be submitted to the Administrative Committee on Standards.

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee contemplates recommending revisions of the following standards. The revised methods, when approved by Committee E-3, will be submitted to the Administrative Committee on Standards.

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[5] The revised recommended practices were accepted by the Society and appear in the 1950 Book of ASTM Methods of Chemical Analysis of Metals.

[6] 1949 Book of A.S.T.M. Standards, Part 2.

Chemical Analysis of Pig Lead (**E 37 - 45**),[3] with the inclusion of the former Photometric Method for the Determination of Bismuth in Pig Lead (E 58 - 45 T),[3]

Chemical Analysis of Lead-and Tin-Base Solder Metal (**E 46 - 49**),[3]

Chemical Analysis of White Bearing Metal Alloys (**E 57 - 49**).[3]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the remaining tentatives, which have stood for two years or more without revision and which are listed below, be retained in their tentative status for further study:

Tentative Recommended Practices for Apparatus for Chemical Analysis of Metals (apparatus for dead-stop end point, mercury cathode cell, and potentiometric titration) (**E 50 - 48 T**).

Tentative Methods for the Polarographic Determination of Lead and Cadmium in Zinc (**E 68 - 46 T**).

ACTIVITIES OF DIVISIONS AND SUBCOMMITTEES

Division F on Ferrous Metals (Arba Thomas, chairman) held one meeting on June 27, 1949, during the Annual Meeting of the Society at Atlantic City. Officers for the ensuing two-year term were elected: Arba Thomas, chairman; J. L. Hague, vice-chairman; W. K. Aites, secretary.

At the meeting held in June, 1949, all of the subgroup chairmen reported that they had completed their review of the present published Standard Methods for Chemical Analysis of Steel (E 30 - 47), of Ferro-Alloys (E 31 - 47), and Electrical Heating Alloys (E 38 - 48). Concrete suggestions for the modification of these procedures were presented. It appears that many of the present published methods can be replaced by entirely new ones that are of comparable accuracy and much less time-consuming. As the subgroups have completed their review assignments satisfactorily, it is planned to reorganize them and set up definite procedures for studying these new techniques. When sufficient satisfactory analytical data have been collected, the new techniques will be recommended for publication as tentative. Many of these new procedures will probably be of the photometric type.

As an immediate result of the thorough review of Methods E 30, E 31, and E 38, Division F is recommending a number of editorial revisions in which an effort has been made, where possible, to eliminate references to alternate procedures within individual procedures, references to routine methods, and footnotes that were considered of an extraneous and sometimes confusing nature. In no instances is it felt that these revisions are changes in the substance of the methods, and it is accordingly recommended that the changes be made editorially before the next printing of the methods.

The division has completed the preparation of proposed new tentative methods for the determination of tin and beryllium in steel.

So far the subgroup responsible for the development of a satisfactory method for the determination of aluminum in stainless steels and similar alloys has been unable to complete a satisfactory method. Two procedures have been studied but neither appears to fulfill the necessary requirements of accuracy.

Division F has been requested to develop a standard A.S.T.M. method for the determination of magnesium (range 0.01 to 0.10 per cent) in nodular iron. A subgroup was appointed and is now working on this problem.

At the June, 1949, meeting of the division, it was the concensus that the current Methods of Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 – 47) were entirely satisfactory for use with malleable iron. This review was made in view of a question raised by Committee A-7 on Malleable Iron Castings.

The program planned for the next few years will keep the membership of Division F very busy, and it is anticipated that the work planned will result in definite improvements in the present published methods.

Division N on Non-Ferrous Metals (Clarence Zischkau, chairman) held one meeting, together with meetings of its subcommittees, at the 1949 Annual Meeting of the Society in Atlantic City. Officers for the ensuing two-year period were elected: Clarence Zischkau, chairman; W. C. Bowden, Jr., vice-chairman; and R. G. Ernst, secretary. Several meetings of local subgroups for special purposes have been held.

In addition to the meeting in June, Subcommittee N-1 on Copper and Its Alloys (R. P. Nevers, chairman) held another meeting in Philadelphia on November 9, 1949. Methods for the determination of sulfur in special brasses and bronzes by both the evolution and combustion methods have been developed. Progress has been made on methods for the analysis of copper-beryllium alloys and for the determination of small amounts of aluminum in special brasses and bronzes. The immediate program is to finish these methods.

Subcommittee N-2 on Lead, Tin, Antimony, Bismuth and Their Alloys (C. L. Luke, chairman) has held a number of local meetings throughout the year. The committee has prepared extensive revisions of the current Methods for Chemical Analysis of Pig Lead (E 37 – 45), Solders (E 46 – 49), and White Metal Bearing Alloys (E 57 – 49). In addition new methods for the chemical analysis of antimony metal and photometric methods for the chemical analysis of lead, tin, antimony, and their alloys have been prepared.

Subcommittee N-3 on Aluminum and Its Alloys (K. C. Braun, chairman) has cooperated closely with Subcommittee N-7 in completing photometric methods for copper, manganese, titanium, chromium, nickel, iron, and bismuth in aluminum and aluminum-base alloys. Work is continuing on methods for small amounts of magnesium and zinc and on proposed photometric methods for silicon.

Subcommittee N-4 on Zinc and Its Alloys (R. K. Bell, chairman) has recommended an optional 8-hydroxyquinoline method for aluminum in zinc-base die-casting alloys. Other revisions in the Standard Methods E 47 – 45 that the subcommittee has under consideration are provision for manganese in the present method for determining lead, an optional method for determining lead, a tannic acid method for determining tin, controlled potential electroanalysis for determining cadmium, and a photometric periodate method for determining manganese.

Subcommittee N-6 on Nickel and Its Alloys (Calvin Sterling, chairman) held a meeting in Newark on October 6, 1949. As a result of this meeting, and following a review of cooperative tests, methods for the chemical analysis of nickel-copper alloys have been completed. A task group under the chairmanship of Mr. G. V. Potter has been at work on methods for metals for electronic uses and has prepared six proposed methods which have been presented to Committee B-4 on Electrical Heating, Resistance, and Related Alloys for their information and review.

Subcommittee N-7 on Photometric Methods (R. G. Ernst, chairman) has been active along the following lines: cooperation with Subcommittee N-3 in preparing photometric methods for the determination of copper, iron, manganese, chromium, nickel, titanium, and bismuth in aluminum and aluminum-base alloys; preparation of a new standard form for the writing of photometric methods, primarily designed to remove ambiguity in connection with "reference solutions" and the use of the term "blank"; and preparation of a revision of the Tentative Recommended Practice for Photometric Methods (E 60 - 46 T) to take care of the new definitions necessitated by the new form for writing methods and to further clarify the term "blank."

Subcommittee N-9 on Magnesium and Its Alloys (V. A. Stenger, chairman) held a meeting in Midland, Michigan, on December 1 and 2, 1949. Methods for copper by the HBr-H_3PO_4 method and for nickel by the dimethylglyoxime-extraction have been completed. A proposed photometric method for aluminum is under consideration. Members of the subcommittee have participated in the collaborative analysis of NBS standard magnesium alloy 171.

Division S on Sampling (J. P. Brull, chairman) held a meeting at the Annual Meeting in Atlantic City in June, 1949. Officers for the ensuing two year period were elected: J. P. Brull, chairman; W. C. Bowden, Jr., vice-chairman; and J. J. Aldrich, secretary. Division S held another meeting on November 9, 1949, at A.S.T.M. headquarters in Philadelphia. At that time two subcommittees were formed: S-1, with E. J. Dunn as chairman, to be concerned with the Sampling of Ferro-Alloys; and S-2, with A. C. Holler as chairman, to be concerned with the Sampling of Non-Ferrous Alloys. A method for the "Sampling of Cast Non-Ferrous Metals and Alloys for Determination of Chemical Composition," prepared by F. M. Barry and R. P. Nevers, was thoroughly discussed and revised. A task group from Division S with representatives from Committees B-2, B-5, B-6, and B-7 will be set up to review this method and complete its preparation during the coming year.

Division G on General Analytical Methods (S. E. Q. Ashley, chairman) held one meeting together with meetings of its subcommittees at Atlantic City in June, 1949, in connection with the Annual Meeting. Officers for the ensuing two-year period were elected: S. E. Q. Ashley, chairman; J. H. Harley, vice-chairman; and H. Kirtchik, secretary. The subcommittees of Division G have been active in the review of methods in preparation for the new edition of the A.S.T.M. Book of Methods of Chemical Analysis of Metals and in revising those sections for which they are directly responsible. Subcommittee G-1 on Reagents (T. C. Bryson, chairman) has prepared a master list of all reagents used in the A.S.T.M. publication with the thought in mind of eliminating different reagents which are used for the same analytical purpose. Subcommittee G-2 on Apparatus (T. L. King, chairman) has prepared specifications on a number of pieces of apparatus and is making some definite proposals for balances. Subcommittee G-3 on Common Procedures (J. H. Harley, chairman) has set up comparisons of procedures for carrying out the same determinations on different materials and has made recommendations for the elimination of minor and nonessential differences. Subcommittee G-4 on Precision and Accuracy (H. B. Lea, chairman) has been cooperating with Committee E-11. A sample of monel metal is being prepared for a statistical study in cooperation with Subcommittee N-6.

Division G will sponsor a symposium on Electroanalytical Methods to be held in Cambridge, Mass., in the fall of 1950.

This report has been submitted to letter ballot of the committee which consists of 102 members; 86 members returned their ballots, of whom 85 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. R. EVANS,
Chairman.

J. W. STILLMAN,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-3 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Chemical Analysis of Nickel-Copper Alloys (E 76 - 50 T),
Chemical Analysis for Sulfur in Special Brasses and Bronzes (E 54 - 50 T),
Chemical Analysis of Antimony Metal (E 86 - 50 T),
Chemical Analysis for Aluminum in Zinc-Base Die-Casting Alloys (E 47 - 50 T), and
Sampling Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (E 88 - 50 T).

New Tentative Photometric Methods for:

Chemical Analysis of Lead, Tin, Antimony and Their Alloys (E 87 - 50 T).

Revision of Tentative Methods for:

Chemical Analysis of Aluminum and Aluminum-Base Alloys (E 34 - 49 T),
Chemical Analysis of Copper-Nickel and Copper-Nickel-Zinc Alloys (E 75 - 49 T).

Revision of Tentative Photometric Methods for:

Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 61 - 46 T) (Redesignated E 35 - 50 T),
Chemical Analysis of Copper and Copper-Base Alloys (E 62 - 46 T), and
Determination of Iron in Slab Zinc (Spelter) (E 64 - 46 T).

Revision of Tentative Recommended Practice for:

Photometric Methods for Chemical Analysis of Metals (E 60 - 46 T).

Tentative Revision of Standard Methods of:

Chemical Analysis of Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (E 30 - 47).

Revision and Reversion to Tentative of Standard Methods of:

Chemical Analysis of Pig Lead (E 37 - 45),
Chemical Analysis of Lead and Tin-Base Solder Metal (E 46 - 49), and
Chemical Analysis of White Metal Bearing Alloys (E 57 - 49).

Withdrawal of Photometric Method for:

Determination of Bismuth in Pig Lead (E 58 - 45 T),
Determination of Iron in (70-30) Copper-Nickel Alloy (E 63 - 46 T), and
Determination of Iron in Lead- and Tin-Base Alloys (E 67 - 46 T).

These recommendations were accepted by the Standards Committee on September 26, 1950, and the new and revised tentatives together with the tentative revision of methods E 30 appear in the 1950 Book of ASTM Methods for Chemical Analysis of Metals.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY[1]

Committee E-4 on Metallography and most of its subcommittees held meetings during the spring meetings of the Society at Pittsburgh, Pa., on February 27 and 28, 1950. At this series of meetings, the electron microscope group, which joined Committee E-4 in 1949 as its new Subcommittee XI, held its first meeting under the sponsorship of Committee E-4. The Committee reported, last year, the possibility of this affiliation. It has been accomplished, and subsequent developments may be noted in detail in the report of this subcommittee, appended hereto under the title "Report of Subcommittee XI on Electron Microstructure of Steel."

The present officers of Committee E-4 were reelected to their present positions.

Committee E-4 continues to show a net growth in membership each year, and is now pleased to report that it has 91 members.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1949 Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method for:

Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (E 79 - 49 T),

Preparing Quantitative Pole Figures of Metals (E 81 - 49 T), and

Determining the Orientation of a Metal Crystal (E 82 - 49 T).

New Tentative Recommended Practice for:

Dilatometric Analysis of Metallic Materials (E 80 - 49 T).

Revision of Tentative Recommended Practice for:

Identification of Crystalline Materials by the Hanawalt X-Ray Diffraction Method (E 43 - 49 T).

These recommendations were accepted by the Standards Committee on December 21, 1949, and the new and revised tentatives appear in the 1949 Book of A.S.T.M. Standards, Part 2.

NEW TENTATIVES

Committee E-4 has in process two new methods which it hopes to submit to the Administrative Committee on Standards shortly after the Annual Meeting of the Society.

The first of these is the proposed Tentative Method of Classification of Ferrite Grain Size, which was reported last year; the second is a proposed Tentative Grain Size Standard for Non-Ferrous Metals (except copper and copper alloys).

While not quite ready for submittal to the Society, Subcommittee VI is nearing completion of a proposed Tentative Method for Determining the Orientation in the Central Portion of Pole Figures.

RECOMMENDATIONS AFFECTING STANDARDS

Because of the large amount of work accomplished in the acceptance of new and revised items last year, there are no recommendations to be made at this time.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

SUBCOMMITTEE ACTIVITIES

Subcommittee II on Definitions (P. A. Beck, chairman) held a meeting in February, 1950, at the spring meetings, at which time it devoted its attention to lists of terms to be defined and abbreviated for Optical Microscopy, X-ray Diffraction, Electron Diffraction, Photography, Grain Size, Alloy Structures, Non-Metallic Inclusion, Powder Metallurgy, Phase Diagrams and Thermal Analysis. Definitions and abbreviations have been prepared relating to Optical Microscopy, X-Ray Diffraction, Grain Size (not complete), Alloy Structures (not complete), and Powder Metallurgy. It is planned to bring the above items to completion, to prepare a list for Electron Microscopy and to proceed with definitions and abbreviations according to the lists already prepared.

Subcommittee III on Nomenclature (P. A. Beck, chairman) has been formed since the last Annual Meeting of the Society, and held its first meeting at Pittsburgh. The membership includes American and English authorities on phase diagrams. Work has been started on exploring the various requirements for phase nomenclature held essential by the members. Initial efforts are in the direction of formulating the basic requirements for a comprehensive system of metallic phase nomenclature. Because of the broad and basic nature of this effort, particular attention is being given to enlist the active cooperation of physical metallurgists both in this country and abroad.

Subcommittee IV on Photography (L. V. Foster, chairman) is aiding Committee E-4 in arranging a photomicrographic exhibit at the Annual Meeting, having the assistance of Subcommittee XI on electron microscope work. As in the past, the judging of the photomicrographs sponsored by Committee E-4 will not be made on the basis of the photograph *per se*, but will be judged on the basis of the entire technique involved in sample selection, preparation, and photography, as well as for the educational value of the techniques involved.

Subcommittee V on Micro-hardness (Alexander Gobus, chairman) held a very active and interesting meeting in Pittsburgh, where round robin test results were thoroughly analyzed and the observed variables brought up for discussion. The influences of types of lighting, loading rates, instrument calibrations, etc., were discussed, as were the advantages of the new stage micrometers of 0.02 μ accuracy, which were developed by Bausch and Lomb Optical Co. at the request of Subcommittee V. Further round robin tests were outlined, and additional checks on reading variations between different observers are planned.

Subcommittee VI on X-ray Methods (W. L. Fink, chairman) is now actively engaged in the investigation of a method for determining the central part of pole figures which was suggested by Shulz of the Institute for the Study of Metals at the University of Chicago. Two members of the subcommittee have constructed specimen holders in accordance with Shulz' suggestion and have started experimental work. The subcommittee is in the process of writing a Tentative Recommended Practice for the Accurate Measurement of Lattice Parameters.

Subcommittee VII on Methods of Thermal Analysis (L. H. Carr, chairman) has reviewed the proposal for the determination of critical points by thermal analysis. This proposal requires but a few minor changes and will then be submitted to letter ballot. This action will probably not be accomplished prior to the Annual Meeting, and the approved proposal will be submitted to the Society at a later date as a revision of Recommended Practice E 14.

Subcommittee VIII on Grain Size (M. A. Grossman, chairman). Section B on Ferrite Grain Size (R. E. Penrod, chairman), has the Proposed Tentative Method of Classification of Ferrite Grain Size in the process of obtaining approval for submission for Society acceptance.

Section C on Non-Ferrous Grain Size (C. H. Samans, chairman) has prepared a Proposed Tentative Grain Size Standard for Non-Ferrous Metals (including both macro and micro grain sizes) which it hopes to have approved by the Society. This new proposal, accomplished with the assistance of other committees interested in nickel, aluminum, magnesium, etc., will go far toward providing a single, universal system of grain counting and of providing classification on standards therefor.

Subcommittee X on Decarburization (J. J. B. Rutherford, chairman).—During the past year, a questionnaire was circulated among the members and others requesting information on "how to measure" decarburization. After an excellent response, the replies were summarized and again circulated to the members. The response to this questionnaire was submitted by the chairman for discussion at the Joint SAE-AISI-ASTM Division XXX meeting held in Detroit, February 21, 1950. At this time, the chairman was asked to prepare a treatise on the various methods of determining decarburization. When prepared, this treatise will be submitted to this subcommittee for discussion and approval.

Subcommittee XI on Electron Microscopy of Steel (G. E. Pellissier, chairman).—During the year 1949, the previously unaffiliated "Joint Committee on Electron Microstructure of Steel" became incorporated with the American Society for Testing Materials as Subcommittee XI on Electron Microstructure of Steel of Committee E-4 on Metallography. This committee originated, more or less spontaneously, during the 1947 Annual Meeting of the Electron Microscope Society of America.

The initial objectives of the committee were as follows:

(*1*) To investigate and evaluate the fidelity and reproducibility of all existing methods of sample and replica preparation.

(*2*) To compare the results of these methods with each other, and with the best obtainable light micrographs on identical structures.

(*3*) To modify or improve existing methods, and to develop new methods, where necessary.

(*4*) To employ the methods found to be most reliable and powerful in Items (*1*), (*2*), and (*3*) for investigating the fine-scale morphology of the decomposition products of austenite and martensite in a plain carbon eutectoid steel.

The initial phase of this work embracing these objectives was completed during 1949, and the results are the subject of the report of this subcommittee appended hereto under the title "Report of Subcommittee XI on Electron Microstructure of Steel."

In full justification of several of the subcommittees which show no report for this year, it must be stated that the thoroughness of the previous year's work, resulting in the Society's acceptance of the numerous Committee E-4 recommendations in 1949, left practically no required activity up until this time.

This report has been submitted to letter ballot of the committee, which consists of 91 members; 71 members returned their ballots, of whom 69 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman

MARY R. NORTON,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-4 presented to the Society through the Administrative Committee on Standards the recommendation that the Methods of Classification of Ferrite Grain Size in Steels be published as tentative. This recommendation was accepted by the Standards Committee on September 26, 1950, and the new tentative methods appear in the 1950 Supplement to Book of ASTM Standards, Part 1, bearing the designation E 89 – 50 T.

ELECTRON MICROSTRUCTURE OF STEEL

FIRST PROGRESS REPORT OF SUBCOMMITTEE XI ON ELECTRON MICROSTRUCTURE OF STEEL, A.S.T.M. COMMITTEE E-4 ON METALLOGRAPHY

This report presents the results to date of studies on the electron microstructure of a eutectoid steel, carried out cooperatively by the Joint Committee on Electron Microstructure of Steel (now Subcommittee XI of Committee E-4). The formation of the committee was instigated by Mr. A. L. Ellis of the International Harvester Co. at the 1947 Annual Meeting of the Electron Microscope Soc. of America. At the time of this meeting, it was felt by interested metallurgists that, although the superior resolving power and magnification of the electron microscope had been utilized to very good advantage in many fields, progress in realizing its potentialities in ferrous metallurgy had been disappointingly slow. Nevertheless, it was the consensus that the inherent advantages of this instrument should be of tremendous value in exploring the fine microstructures of steel, such as bainite, martensite, and tempered martensite, which are beyond the resolving power of the light microscope. It was believed, furthermore, that information of this kind would be extremely useful in advancing knowledge of the relationships between the morphology of these structures and the mechanical properties.

In the preceding years, the electron microscope had been used in such studies in individual laboratories to a certain extent. However, when the results of these isolated investigations were compared, the electron micrographs of presumably similar structures were so different, and in many cases were so dissimilar to the light microstructures, as to preclude any consistent interpretations and conclusions. It was suspected that this confusion was largely a reflection of complications introduced by the many new variables involved in the techniques of electron metallography, such as difficulties in the preparation and etching of specimens for these high magnifications, the wide variety of replica techniques, the inherent low contrast of these replicas, and difficulties arising from the more complex photographic procedure involved. It was reasoned that a group of several laboratories, diligently applying a few of the most promising techniques to identical structures, should be able to determine what degree of consistency could be achieved among electron micrographs—first of all, on those microstructures which could be resolved with the light microscope, and therefore, could be compared directly, and finally, on those microstructures that are not resolvable by light microscopy. This line of reasoning served as the basis for the formation of the Joint Committee on Electron Microstructure of Steel. This committee was formally organized in Pittsburgh, Pa., on February 13, 1948, and consisted of the following individuals and organizations:

A. L. Ellis—Manufacturing Research Dept., International Harvester Co.
V. G. Peck—Research Lab., United States Steel Corp.
R. D. Heidenreich—Bell Telephone Labs.
C. M. Schwartz—Battelle Memorial Inst.
D. M. Teague—Research Division, Chrysler Corp.
W. L. Grube—Research Laboratories Division, General Motors Corp.
S. B. Maloof—Division of Metallurgy, Pennsylvania State College
C. F. Tufts—Armour Research Foundation
M. Baeyertz—Armour Research Foundation
J. M. Hodge—Research and Development Division, Carnegie-Illinois Steel Corp.
G. E. Pellissier—Research Lab., Carnegie-Illinois Steel Corp.

Since that time, this committee has grown larger and some changes in membership have occurred. The policy has been that each cooperating laboratory be represented on this committee by an electron microscopist and a metallurgist. In August, 1949, this committee became affiliated with the American Society for Testing Materials' Committee E-4 on Metallography, and is designated as Subcommittee XI on Electron Microstructure of Steel. The present membership of this Subcommittee is as follows:

G. E. Pellissier, *Chairman*—Carnegie-Illinois Steel Corp.
D. M. Teague, *Secretary*—Chrysler Corp.
W. L. Grube—General Motors Corp.
E. J. Dofter—Chrysler Corp.
J. M. Hodge—Carnegie-Illinois Steel Corp., Pittsburgh, Pa.
J. R. Vilella—United States Steel Corp., Research Lab., Kearny, N. J.
R. M. Fisher—United States Steel Corp., Research Lab., Kearny, N. J.
C. M. Schwartz—Battelle Memorial Inst., Columbus, Ohio
A. L. Ellis—International Harvester Co., Chicago, Ill.
C. F. Tufts—Armour Research Foundation, Chicago, Ill.
W. F. Craig—Armour Research Foundation, Chicago, Ill.
E. F. Fullam—Knolls Research Lab., General Electric Co., Schenectady, N. Y.
L. R. Cooper—Heppenstall Co., Pittsburgh, Pa.
H. C. O'Brien—Heppenstall Co., Pittsburgh, Pa.
L. S. Birks—Naval Research Lab., Washington, D. C.
P. Coheur—Centre Belge De Metallurgie Physique, 12 Quai Paul Van Hoegaerden, Liege, Belgium
L. Habraken—Centre National De Recherches Metallurgiques, Liege, Belgium

The research program, the results of which are the subject of this report, was formulated at the February 13, 1948, meeting. Essentially, this plan consisted of heat treating a series of pieces of a eutectoid steel to specific microstructures, and dividing these pieces into specimens which were supplied to each cooperating laboratory for investigation by means of electron microscopy. The composition of the steel used was as follows:

Carbon, per cent	0.87
Manganese, per cent	0.44
Silicon, per cent	0.17
Phosphorus, per cent	0.013
Sulfur, per cent	0.006
Nickel, per cent	0.39
Chromium, per cent	0.21

All of the heat treatments were carried out at the United States Steel Research Lab.; the heat treatments and resulting microstructures are described in Table I. Light micrographs of each of these structures were prepared at the United States Steel Research Lab. at a magnification of 2500. These light micrographs, which are reproduced in this report, are believed to be representative of the best metallographic techniques currently available.

The program for the electron microscope studies included some variations in metallographic polishing and etching practices, replica techniques, and photographic procedures. Both mechanical polishing and electropolishing were used to some extent, but mechanical polishing was used predominantly in obtaining the results reported here. The etching reagents employed in this work were, picral, nital, and ferric chloride - hydrochloric acid. Three replica techniques were investigated, namely, formvar, parlodion, and polystyrene-silica. Most of the formvar and parlodion replicas were shadow-cast either with chromium, platinum, or palladium to enhance the contrast. In general, the electron micrographs were made at an initial magnification of 5000, and then were enlarged to a final magnification of 15,000. In addition to these high magnification micrographs, electron micrographs of these structures were prepared at an initial magnification of 750, and were enlarged to 2500, in order to permit comparison with the light micrographs. For the most part, the enlarged prints were made directly from the photographic negatives, but in a few instances, reverse prints were made. Information pertaining to the electron microscope techniques and procedures is presented in detail in the Appendix to this report.[1]

TABLE I.—STRUCTURES INVESTIGATED AND CORRESPONDING HEAT TREATMENTS.

Specimen	Structure	Heat Treatment Following Austenitizing[a]	Rockwell Hardness (C Scale)
No. 1	1300 F. coarse pearlite	3½ hr. at 1300 F.	13.8
2	1100 F. fine pearlite	25 sec. at 100 F.	38.5
3	950 F. fine pearlite and bainite	6 sec. at 550 F., 60 sec. at 950 F.	41.0
4	750 F. bainite	6 sec. at 550 F., 7 min. at 750 F.	43.8
5	500 F. bainite	2½ hr. at 500 F.	57.4
6	martensite (as-quenched)	as-quenched in brine	65.1
7	400 F. tempered martensite	brine quenched, tempered at 400 F. for 1 hr.	60.1
8	600 F. tempered martensite	brine quenched, tempered at 600 F. for 1 hr.	53.4
9	800 F. tempered martensite	brine quenched, tempered at 800 F. for 1 hr.	44.1
10	1100 F. tempered martensite	brine quenched, tempered at 1100 F. for 1 hr.	32.6
11	1250 F. spheroidized carbides	brine quenched, tempered at 1250 F. for 12 hr.	20.1
12	500 F. bainite (partial transformation)	15 min. at 500 F.	......

[a] All of these specimens were austenitized at 1800 F. for 35 min. and were air cooled from the final indicated temperature.

From the large number of electron micrographs accumulated in this program, the series reproduced in this report have been selected as being most representative of the microstructures studied, and of the various techniques employed. The micrographs pertaining to these different microstructures are separately discussed in some detail below.

COARSE PEARLITE

Isothermal Transformation at 1300 F.

The light and electron micrographs of coarse pearlite are shown in Figs. 1 to 8. The electron microstructure of coarse pearlite, at a magnification of 2500, is shown in Fig. 3. The close similarity between the appearance of this electron micrograph and that of the light micrographs shown in Figs. 1 and 2, is evident. The fact that the shades are reversed in the electron micrograph, so that the cementite lamellae appear light and the ferrite matrix, dark, is an inherent characteristic of the single-stage, negative replica technique. With this technique, the areas of the etched metal surface which stand in relief—in this instance cementite lamellae—are reproduced in the replica as thinner sections, and, since electron transmission is greater in these regions, they appear lighter in the electron image.

Figure 4 is an electron micrograph at 2500 of the same structure, but etched with $FeCl_3$-HCl instead of picral. It is evident from this micrograph that the $FeCl_3$-HCl etch has severely attacked the ferrite matrix in the pearlite colonies at the lower right of the micrograph, causing excessive relief of the cementite lamellae in these regions; this results in a somewhat distorted picture of the structure in these colonies in that the areas corresponding to the cementite lamellae are artificially enlarged. The fact that this etchant attacks the ferrite in some pearlite colonies more severely than in other colonies is a characteristic of this etchant; it has been pointed out previously in the metallurgical literature that the attack on ferrite by this etchant is of a crystallographic nature and that the rate of attack is dependent on the orientations of the ferrite in the various pearlite colonies which are exposed in the specimen surface. This type of etch is useful for clearly delineating pearlite colonies, but it would not be suitable for preparing electron micrographs for measurement of interlamellar spacing in coarse pearlite.

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[1] See p. 489.

This same structure is shown in the electron micrograph of Fig. 5 at a magnification of 15,000; again, the similarity to the light micrographs is quite obvious. It will be noted that the relative amounts of ferrite and cementite are approximately in accordance with the theoretically expected ratio. Some detail can be observed in the ferrite matrix, such as scratches and general roughening remaining from the mechanical polishing of the sample. The cementite lamellae are clearly defined, and the serrations at the edges of the lamellae, presumably caused by mechanical abrasion during polishing, are also apparent. Electron micrographs of this kind would be suitable for making precise measurements of the interlamellar spacings of pearlite. Figures 6 and 7 illustrate the effect of replica shadowing at two different angles, 45 and 30 deg. to the surface, respectively, on the appearance of the electron micrographs of coarse pearlite. In this instance, the unshadowed replica (Fig. 5) had adequate contrast so that very little is gained by shadowing; however, for the finer structures discussed below, shadowing was found to be essential in order to obtain the desired contrast. A comparison of Figs. 5 and 6 will show that shadowing at the high angle has not perceptibly changed the appearance of the pearlite; the shape, size, and spacing of the cementite lamellae are strictly comparable in these two micrographs. The effect of shadowing at a low angle, as shown in Fig. 7, is to effectively enlarge the regions of the replica corresponding to the cementite lamellae, so as to give a somewhat distorted impression of the relative amounts of the two phases present. Electron micrographs, such as Fig. 7, would not be suitable for making precision measurements of the interlamellar spacing of pearlite. In view of this possibility of dimensional distortion at a low angle of shadow casting, most of the electron micrographs selected for use in this report were made from replicas shadowed at a relatively high angle. The electron micrograph of Fig. 8 shows this structure at a magnification of 15,000, as etched with $FeCl_3$-HCl. This micrograph illustrates quite clearly the crystallographic nature of the attack on ferrite by $FeCl_3$-HCl; in the pearlite colony at the left of the micrograph, this etchant has severely attacked the ferrite and has developed crystal facets, whereas in the colony at the right the attack is milder and more uniform.

FINE PEARLITE

Isothermal Transformation at 1100 F.

The light and electron micrographs of fine pearlite at 2500× are shown in Figs. 9 and 10, respectively. In the electron micrograph, all of the pearlite lamellae, irrespective of their apparent spacing (orientation with respect to the specimen surface), are clearly resolved at this magnification. This is in marked contrast to the light micrograph in which less than 50 per cent of the area of the field is perceptibly resolved. This is a striking example of the superior inherent resolving power of the electron microscope, even at comparatively low magnifications.

This same structure, etched with $FeCl_3$-HCl, is shown at a magnification of 15,000 in the electron micrograph of Fig. 11. This micrograph presents about as clear a picture of fine pearlite as could be expected, and it would be possible to make precise statistical measurements of the interlamellar spacing on micrographs such as this. Furthermore, since all of the pearlite is resolved in the electron micrograph, this technique has a decided advantage over light microscopy in that it provides assurance that no structures other than pearlite (for instance, bainite or martensite) are present. This micrograph was chosen, from the many prepared, as being most representative of the structure of fine pearlite; the very closely spaced cementite lamellae are perfectly resolved, and the several pearlite colonies are readily distinguished. In the field of this micrograph, the cementite lamellae appear to intersect the specimen surface almost perpendicularly, so that the observed interlamellar spacing is nearly equal to the true spacing. The electron micrographs of Figs. 12 and 13 show other areas of this specimen where the cementite lamellae intersect the surface more obliquely. In the areas in which this obliquity is most pronounced, the effects of the severe attack by the $FeCl_3$-HCl etchant and of the shadow-casting have combined to create a "snow-drift" appearance at the edges of the cementite lamellae.

FINE PEARLITE AND BAINITE

Isothermal Transformation at 950 F.

The electron microstructure of this specimen, as etched with $FeCl_3$-HCl is shown at a magnification of 2500 in Fig. 15. Although the resolution in this micrograph is distinctly superior to that in the corresponding light micrograph (Fig. 14), the very fine structure is not well delineated at this magnification. However, the pearlite in this specimen is very fine, and although the electron microscope is capable of resolving the lamellae, the magnification is not sufficiently high to permit photographic and visual resolution.

At a magnification of 15,000, however, the very fine details of this microstructure are fully resolved, as shown in Fig. 16. In this micrograph, two types of structure are discernable, that is, areas of resolved, very fine pearlite, similar to that discussed above, and regions in which the cementite platelets are larger and more randomly distributed and oriented than in pearlite; the latter areas can be seen in the upper right-hand, and lower center of Fig. 16. As will be evident from a comparison with the electron micrographs of upper bainite discussed below, there is reason to believe that this second structure is, in fact, upper bainite. Although the dual nature of the structure of this specimen is suggested by both the light and electron micrographs at 2500×, neither the pearlite nor bainite is well enough resolved to permit positive identification. However, in this electron micrograph at 15,000×, the shape, size, and arrangement of cementite particles are revealed within the bainite needles, the outlines of which are just perceptible in the micrographs at 2500×. Examination of the band-shaped area of bainite in the upper right-hand corner of Fig. 16, reveals that, within a bainite needle, the cementite platelets are, in general, rather long, and are roughly oriented with their long dimension parallel to

the axis of the **needle. It** will be noted **that** the very fine **pearlite** formed at this **temperature** is not as well organized as are the pearlites formed at the higher temperatures. It is rather surprising to find that, even though the gross appearance of the bainite is quite different from that of the pearlite, these two structures both consist of cementite platelets in a ferrite matrix; the principal difference is of degree, the cementite platelets in the bainite being shorter, chunkier, and their distribution and orientation are less regular.

BAINITE

Isothermal Transformation at 750 F.

The acicular nature of the microstructure of this specimen is evident in both the light micrograph (Fig. 17) and in the electron micrograph (Fig. 18), at a magnification of 2500. Although the gross features of this structure are more clearly delineated in the electron micrograph than in the light micrograph, the very fine details of the structure are not adequately resolved at this magnification.

On the other hand, the electron micrograph at 15,000× (Fig. 19) clearly reveals the ultimate microstructure of this upper bainite; the sharply defined cementite platelets shown in this micrograph make a particularly marked contrast to the almost completely unresolved structure shown by the corresponding light micrograph at 2500×. The cementite platelets in this bainitic structure, formed at 750 F., seem to be characteristically chunky and more or less randomly oriented and distributed in the ferrite matrix. The similarity of this structure to the presumably bainitic portion of the structure formed at 950 F. is evident, but the 750 F. structure is generally less well organized, and the cementite platelets are more randomly distributed. Most of the cementite platelets are large and chunky (predominantly 10,000 Å in length), but there are also visible, many very small cementite particles of the order of 600 Å.

BAINITE

Isothermal Transformation at 500 F.

The microstructure of this specimen at a magnification of 2500 is shown in the light micrograph of Fig. 20 and in the electron micrograph of Fig. 21. The acicular nature of this lower bainite is suggested by both of these micrographs; however, the coarse features of the microstructure are quite clearly resolved in the electron micrograph, whereas in the light micrograph, none of the structure is perceptibly resolved.

The ultimate microstructure of lower bainite, formed at 500 F., is revealed in the electron micrograph at 15,000 of Fig. 22. As in the case of upper bainite, this structure consists of cementite platelets in a ferrite matrix. It is characterized by ferrite bands containing many small cementite platelets in parallel array; within each band, the platelets are oriented at the same angle to the axis of the band. In general, these bands are outlined by relatively large, chunky particles of cementite. Reference to the electron microstructure at 2500× strongly suggests that these bands are the bainite needles observed at the lower magnification, and that the large, chunky cementite particles delineate the needle boundaries. These bainite needles are shown even more strikingly in the electron micrographs at 15,000× of Figs. 23 and 24. It is clearly evident from these micrographs that the small, oriented cementite platelets are, in fact, situated within these bainite needles; in Fig. 23, the principal plane of the cementite platelets is nearly parallel to the specimen surface, whereas in Fig. 24, it generally intercepts the surface at a large angle.

MARTENSITE

Brine Quenched

The light and electron micrographs at 2500× of as-quenched martensite are shown in Figs. 25 and 26, respectively. Both of these micrographs show the acicular structure characteristic of martensite, but no significant detail is visible in the electron micrograph beyond that observed in the light micrograph.

The electron micrograph of this same specimen, etched with 2 per cent nital plus zephiran chloride, is shown at a magnification of 15,000 in Fig. 27. In this micrograph, geometrically-shaped areas of retained austenite are clearly revealed, standing in relief above the matrix of intersecting martensite needles. Although this etchant evidently has caused a general roughening and pitting of the martensite needles, no additional structural detail of significance is visible within the needles. The fact that even the very small areas of retained austenite are so clearly delineated suggests that this technique would be of value in improving the accuracy and sensitivity of metallographic determinations of retained austenite.

TEMPERED MARTENSITE

1 hr. at 400 F.

The light and electron micrographs of this structure, at a magnification of 2500, are shown in Figs. 28 and 29, respectively. Very little significant information is revealed by either of these micrographs at this magnification, but, in the electron micrograph, the areas of retained austenite and tempered martensite are much more clearly delineated. Comparison of the electron micrograph of this structure with that of Fig. 26, shows that no change in the coarse features of the as-quenched martensitic structure has occurred on tempering at 400 F. for 1 hr.

Figure 30 is an electron micrograph at 15,000× of this same specimen, etched with 2 per cent picral plus zephiran chloride. The structure shown in this micrograph differs from that of the untempered martensite (Fig. 27) principally in that fine detail is now visible in the tempered martensite needles. Although this structure is extremely fine and not clearly resolved, its appearance suggests that discrete particles have precipitated on tempering at 400 F. The appearance of the structure at the upper right-hand corner of this micrograph is different from that of the tempered martensite and retained austenite and is suggestive of the structure of lower bainite, as previously shown.

TEMPERED MARTENSITE

1 hr. at 600 F.

The light and electron micrographs at 2500× of this structure are shown in Figs. 31 and 32, respectively. Although the light micrograph suggests that discrete carbide platelets have precipitated at this tempering temperature, these platelets are much more clearly resolved in the electron micrograph.

The ultimate microstructure of this 600 F. tempered martensite is clearly shown in the electron micrograph at 15,000× of Fig. 33. In this micrograph, the small, discrete carbide platelets are plainly evident in the ferrite matrix; many of these particles are as small as 300 Å. These platelets show some tendency toward parallel alignment in individual regions, suggesting an orientation relationship with the parent martensite needles. It is interesting to note that this structure is quite similar in many respects to that observed in the specimen of 750 F. bainite (Fig. 19).

The platelets in the upper bainite, however, are larger, chunkier, and are more randomly distributed and oriented in the matrix. The electron microstructure of this same specimen is shown in another electron micrograph at 15,000× (Fig. 46), but in this particular field, there are a few areas, indicated by arrows, which are very similar in appearance to the structure observed for lower bainite. This suggests that these regions are in fact 600 F. bainite resulting from transformation of retained austenite during tempering.

TEMPERED MARTENSITE

1 hr. at 800 F.

The light and electron micrographs at 2500× of this structure are shown in Figs. 34 and 35, respectively. As in the case of the 600 F. tempered martensite, the presence of discrete carbide platelets is suggested by the light micrograph, but they are clearly evident in the electron micrograph. Even in this electron micrograph, however, only the largest particles are clearly visible at this magnification.

This structure is shown at a magnification of 15,000 in the electron micrograph of Fig. 36. It is quite similar to that of the 600 F. tempered martensite, except that considerable coalescence of the carbide particles obviously has occurred, so that they are larger and chunkier, and noticeably fewer of the very fine particles are present. Furthermore, the rounded appearance of some of the particles indicates that some spheroidization has occurred at this stage of tempering. In the case of this structure particularly, electron microscopy has a decided advantage over light microscopy in that it permits accurate measurement of the particle size and distribution, and observation of the degree of spheroidization. Here again, the similarity of this structure to the 750 F. bainite is notable, the principal distinction being the relatively large number of very small particles in the upper bainite structure.

TEMPERED MARTENSITE

1 hr. at 1100 F.

The light and electron micrographs of this structure are shown at a magnification of 2500 in Figs. 37 and 38, respectively. The superior resolution achieved with the electron microscope is demonstrated particularly well by a comparison of these two micrographs. This superior resolution makes it possible, even at this relatively low magnification, to evaluate the degree of spheroidization, and the actual distribution of the discrete carbide particles in this tempered martensitic structure. A concentration of relatively large carbide particles, in locations which seem to correspond with the prior austenite grain boundaries, is evident in this micrograph, and the arrangement of carbide particles within these prior austenite grains suggests some relationship to the original acicular structure of the martensite.

The electron microstructure at a magnification of 15,000 is shown, beautifully delineated, in the electron micrograph of Fig. 39. It is clearly evident from this electron micrograph that spheroidization of the carbide particles is nearly complete and that coalescence has progressed much farther than at 800 F. The predominant carbide particle size is about 2500 Å, but there are several particles as small as 600 Å, and some as large as 10,000 Å. Another electron micrograph at 15,000 × of this structure (Fig. 47) shows, very strikingly, the concentration of coarse carbide particles at prior austenite grain boundaries, which was noted in the

2500 × electron micrograph (Fig. 38). The clear picture of the size, shape, distribution and degree of spheroidization of the carbide particles furnished by these electron micrographs is in marked contrast to the rather poorly defined structure shown in the light micrograph.

TEMPERED MARTENSITE

12 hr. at 1250 F.

The light and electron micrographs of this spheroidized structure at a magnification of 2500 are shown in Figs. 40 and 41, respectively. In this instance, the similarity between the light and electron micrographs is very pronounced, and this similarity, as in the case of coarse pearlite, provides good assurance that the replica technique employed in these electron microscope studies does, in fact, present a true, high fidelity picture of the microstructure.

These spheroidized carbides are shown at a magnification of 15,000 in Fig. 42. Little additional information is gained from this electron micrograph, but the fine carbides are clearly revealed at this magnification, so that accurate particle size distribution measurements could be made from such electron micrographs. Furthermore, there is some evidence in this electron micrograph of a boundary structure in the ferrite matrix; although this is not pronounced, it suggests that this technique might be useful in studying structural changes in the ferrite during tempering.

BAINITE

Partial Transformation at 500 F.

This specimen was added to the program in the early stages of this work, on the basis that, with light microscopy, the martensite provides a better background contrast for the bainite needles. The light and electron micrographs of this partially transformed structure at a magnification of 2500 are shown in Figs. 43 and 44, respectively. It is evident from a comparison of these micrographs that, with the electron microscope technique, the contrast between the bainite and the martensite is not nearly so pronounced; thus, the expected advantage is not realized, and, in fact, it now seems that, with present techniques, more can be learned from the fully transformed structures.

The electron microstructure at 15,000 × of this partially transformed specimen is shown in Fig. 45. The bainitic areas, indicated by arrows, are similar in structure to that of lower bainite as previously shown in Figs. 22 to 24. As in the case of the specimen of as-quenched martensite, the areas of retained austenite in the martensite matrix are clearly delineated, but no significant detail is visible in the martensite needles of this partially transformed bainite specimen.

SUMMARY AND CONCLUSIONS

The results of this work to date demonstrate, first of all, that electron microscope replica techniques are capable of yielding true, high fidelity pictures of steel microstructures. This is evidenced particularly by the close similarity in appearance between electron micrographs and light micrographs of coarse pearlite and coarse spheroidite. Also, it has been shown that the extraordinary resolving power and magnification of the electron microscope can be utilized to very good advantage to investigate those fine microstructures which cannot be adequately defined by means of the light microscope. Furthermore, even in the case of the coarse microstructures, which are resolved by the light microscope, additional fine structural detail may be revealed by the electron microscope. The sharp, high-magnification electron micrographs also permit more accurate measurement of structural dimensions than can be obtained from the best light micrographs. These features of the electron microscope undoubtedly will be of great value in metallurgical studies of (1) the precise nature and ultimate microstructure of austenite and martensite decomposition products, (2) the mechanisms of formation of these decomposition products, and (3) the relationships between microstructure and properties of steel.

More specifically, some of the pertinent findings of this application of the electron microscope to the investigation of steel microstructures are as follows:

1. In specimens of eutectoid steel transformed at 1100 F., portions of the field which have hitherto been unresolved by the light microscope are shown by the electron microscope to be, in fact, pearlite of very small interlamellar spacing.

2. It has been found that eutectoid steel transformed at 950 F. contains two distinctly different microstructures, namely, very fine pearlite, and upper bainite.

3. The microstructure of the upper bainite formed at 750 F. has been completely resolved and has been found to consist of discrete, chunky, cementite platelets, more or less randomly distributed and oriented in the ferrite matrix. The predominant length of these platelets is 10,000 Å, but there are also many small cementite particles of the order of 600 Å.

4. The microstructure of lower bainite formed at 500 F. has been completely resolved and has been found to consist of ferrite bands, containing small cementite platelets in parallel array; these bands are outlined by relatively large, chunky, carbide platelets.

5. In brine-quenched martensite, even the very small areas of retained austenite are clearly delineated, which suggests that this technique would be of value in improving the accuracy and sensitivity of quantitative metallographic determinations of retained austenite.

6. Although the structure of martensite tempered at 400 F. is extremely fine and has not been clearly resolved, the appearance of the electron micrographs suggests that carbide particles have started to precipitate at this temperature.

7. The microstructure of martensite tempered at 600 F. has been completely resolved and has been found to consist of small, discrete, carbide platelets in a ferrite matrix; many of these particles are as small as 300 Å.

8. The microstructure of martensite tempered at 800 F. has been found to be quite similar to that of the 600 F. tempered martensite, except that considerable coalescence of the carbide particles has occurred, and there is evidence that spheroidization has begun at this temperature.

9. The electron microstructure of martensite tempered at 1100 F. shows that spheroidization of the carbide particles is essentially complete at this temperature, and that coalescence of the carbide particles has progressed considerably beyond that observed at 800 F.

FUTURE WORK

During 1950 and 1951, Subcommittee XI plans to investigate the following phases of this work on eutectoid steel more intensively:

1. The transition in microstructure from fine pearlite to upper bainite, which occurs upon isothermal decomposition of austenite within the temperature range 950 to 750 F.

2. The transition in microstructure from upper bainite to lower bainite,

which occurs upon isothermal decomposition of austenite within the temperature range 750 to 550 F.

3. The changes which occur in the microstructure of martensite during the initial stages of tempering at temperatures below 600 F.

4. The microstructure of decomposition products which first form from retained austenite on tempering at various temperatures.

Further, long-range plans for electron microscope investigation of the microstructure of eutectoid steel include the following items:

1. Study of the changes in microstructure which occur in lower bainite, as isothermal decomposition of austenite to lower bainite proceeds to completion.

2. Study of the microstructure of isothermal decomposition products of austenite which form below the M_s temperature and comparison with the microstructure of martensite formed on quenching.

3. Statistical measurements of the carbide particle size distribution in the bainitic structures, and comparison with similar measurements on martensites tempered at temperatures corresponding to those at which the bainites were formed.

4. Correlation of the electron microstructure of the various decomposition products of austenite and martensite with the mechanical properties of these structures.

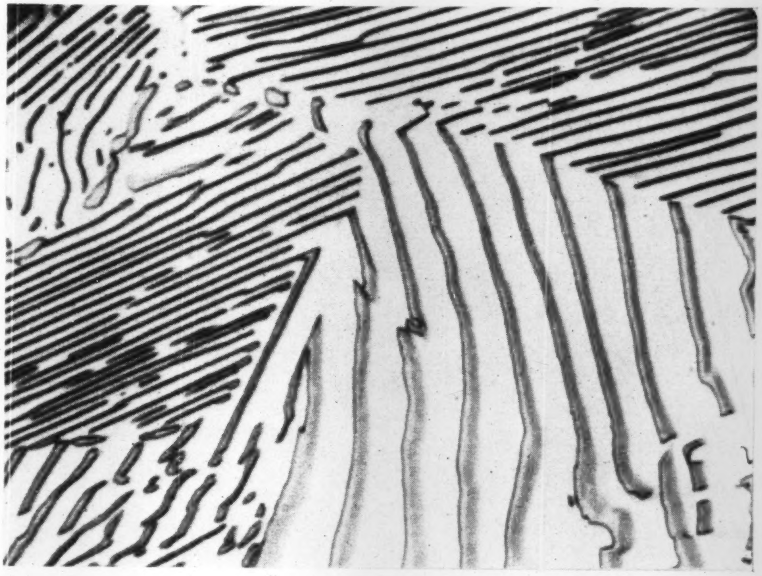
Respectfully submitted on behalf of the subcommittee,

G. E. PELLISSIER,
Chairman.

D. M. TEAGUE,
Secretary.

FIG. 1.—Light Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



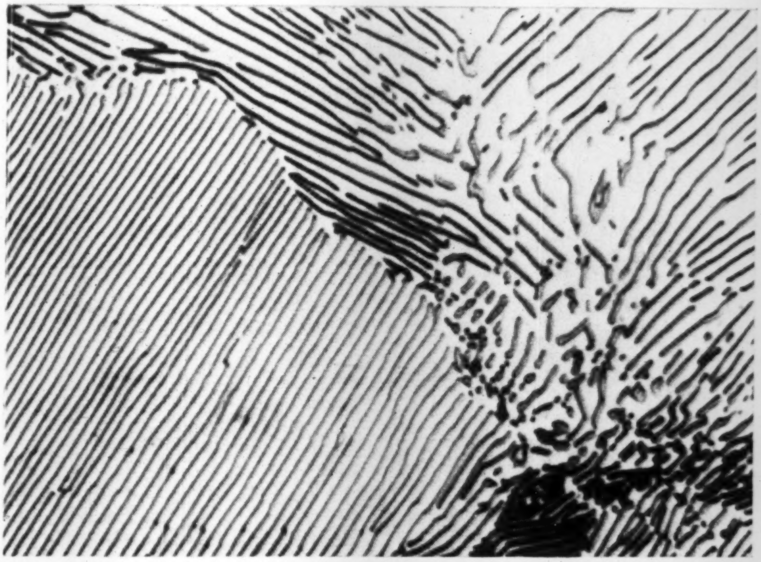
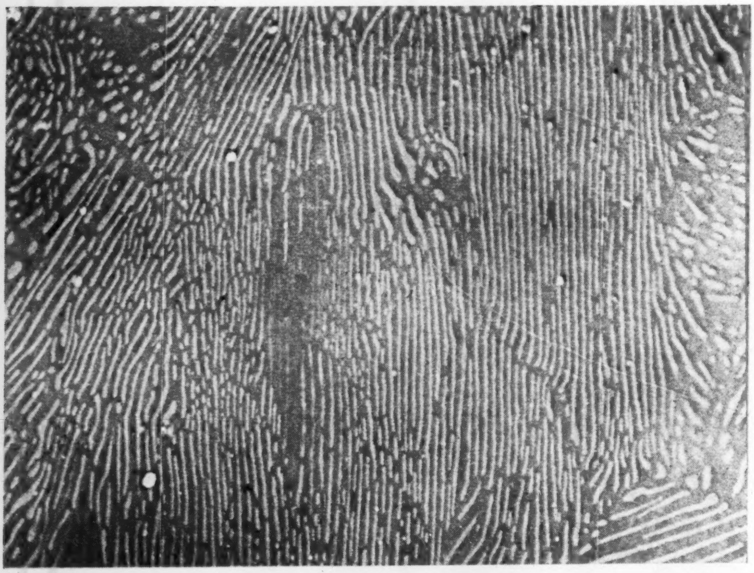


FIG. 2.—Light Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.

FIG. 3.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., Picral Etch, Formvar Replica, Palladium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.



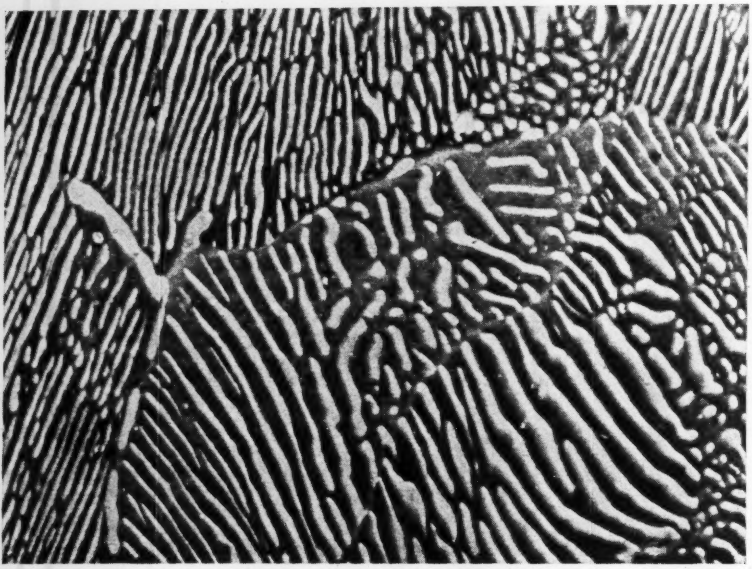


FIG. 4.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., $FeCl_3$-HCl Etch, Formvar Replica, Unshadowed (× 2500).

Courtesy of Chrysler Corp.

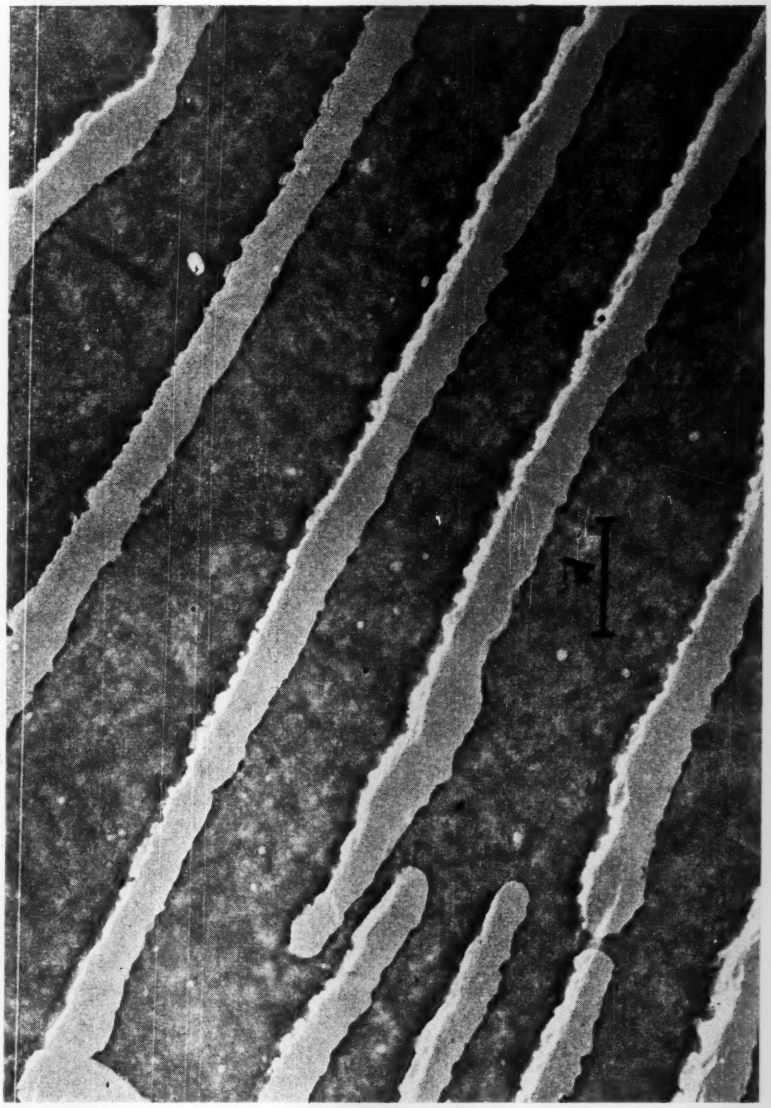


FIG. 5.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., Picral Etch, Formvar Replica, Unshadowed (× 15,000).

Courtesy of General Motors Corp.

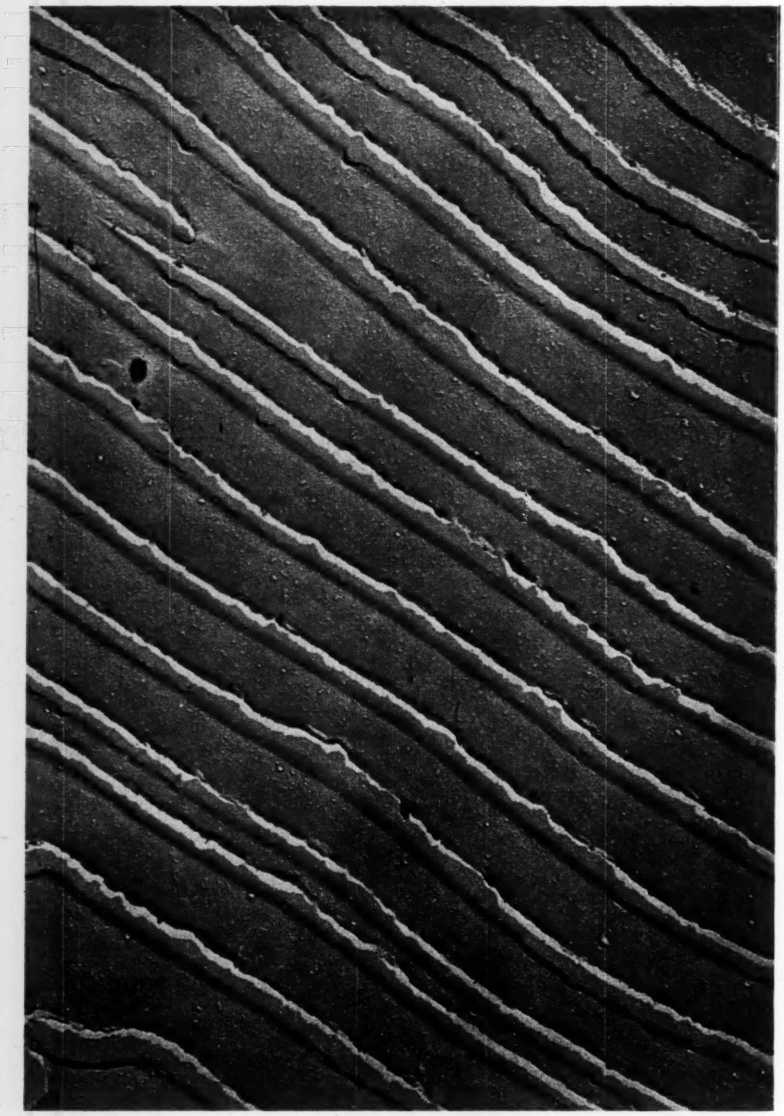


FIG. 6.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., Picral Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

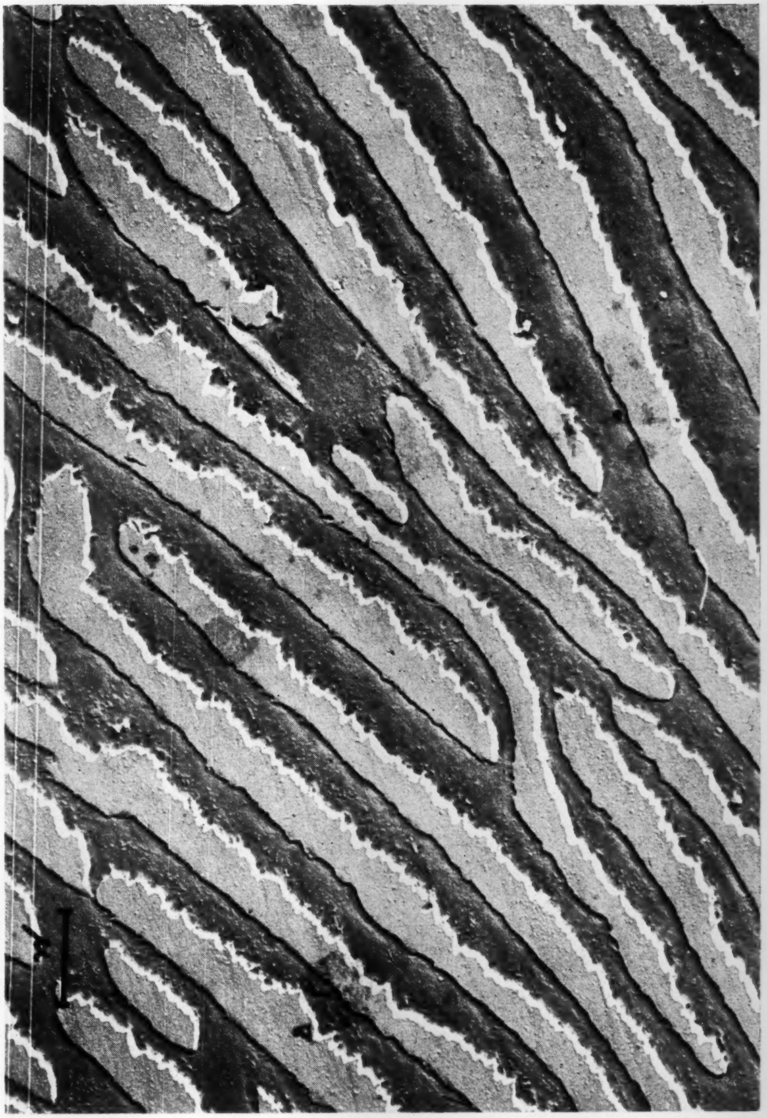


FIG. 7.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., Picral Etch, Parlodion Replica, Chromium Shadowed at 27 deg. (× 15,000).

Courtesy of General Motors Corp.

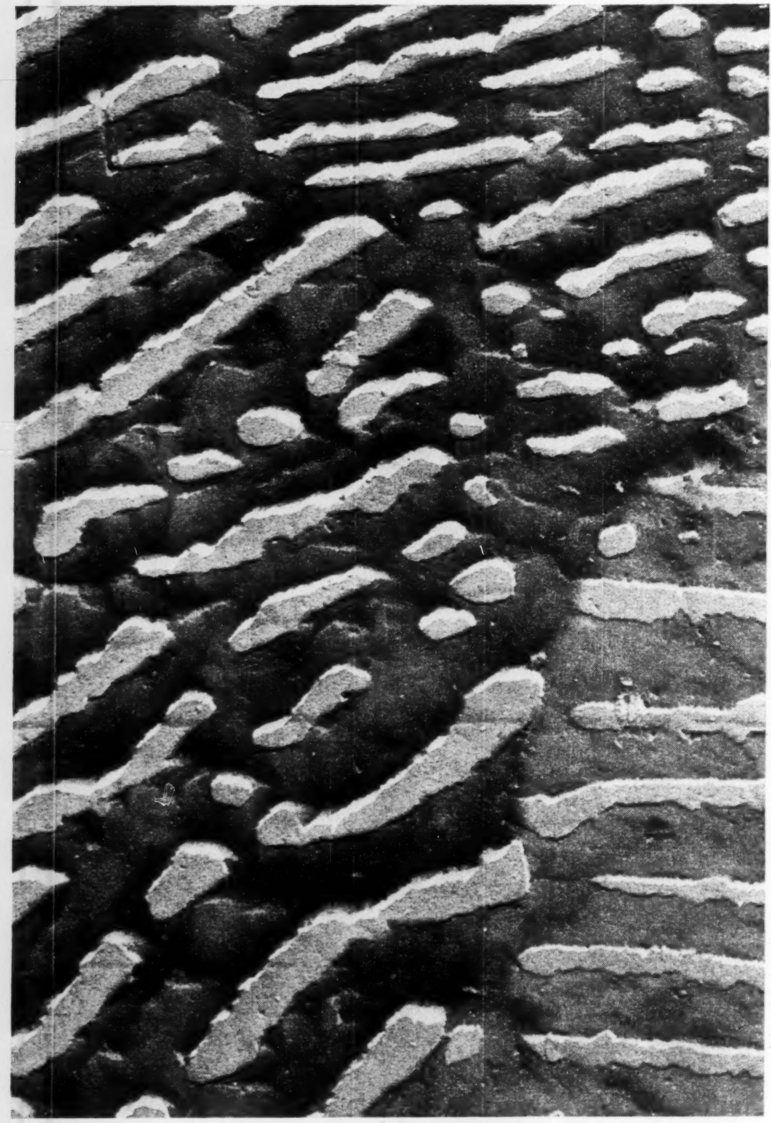
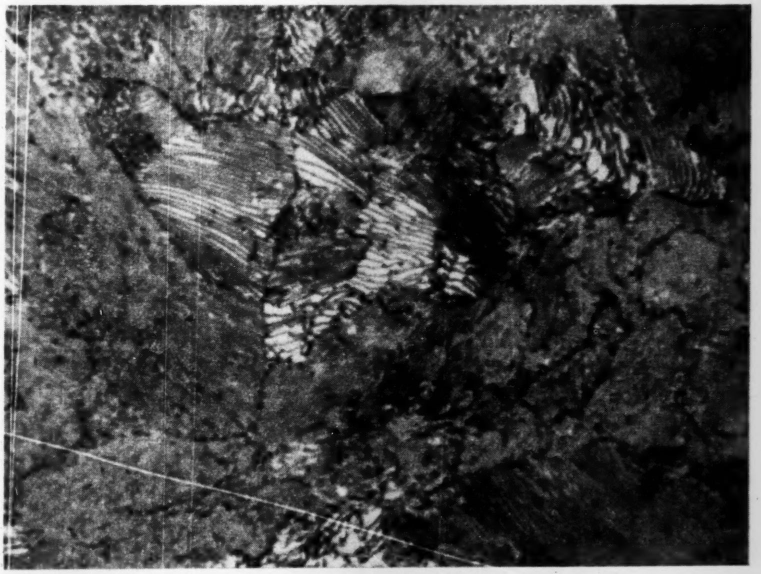


FIG. 8.—Electron Micrograph of Coarse Pearlite, Isothermal Transformation at 1300 F., $FeCl_3$-HCl Etch, Formvar Replica, Chromium Shadowed at 27 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 9.—Light Micrograph of Fine Pearlite, Isothermal Transformation at 1100 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



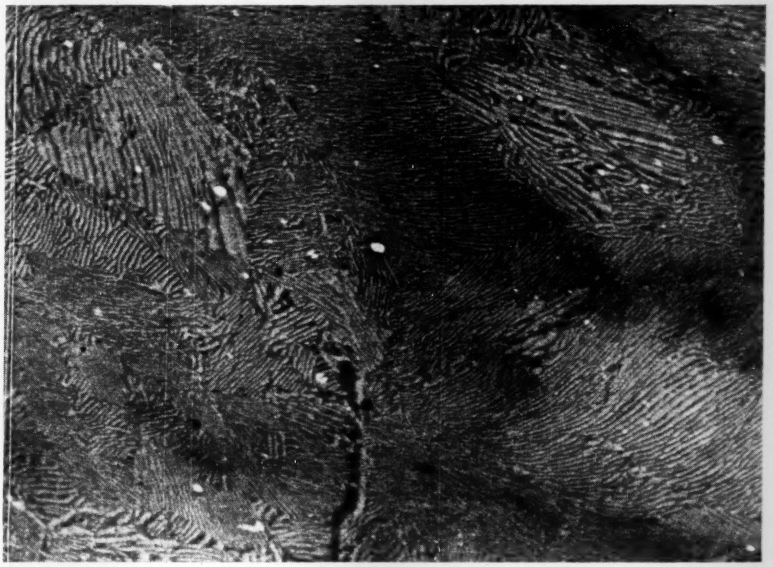


FIG. 10.—Electron Micrograph of Fine Pearlite, Isothermal Transformation at 1100 F., Picral Etch, Formvar Replica, Palladium Shadowed at 27 deg. (× 2500).

Courtesy of Chrysler Corp.

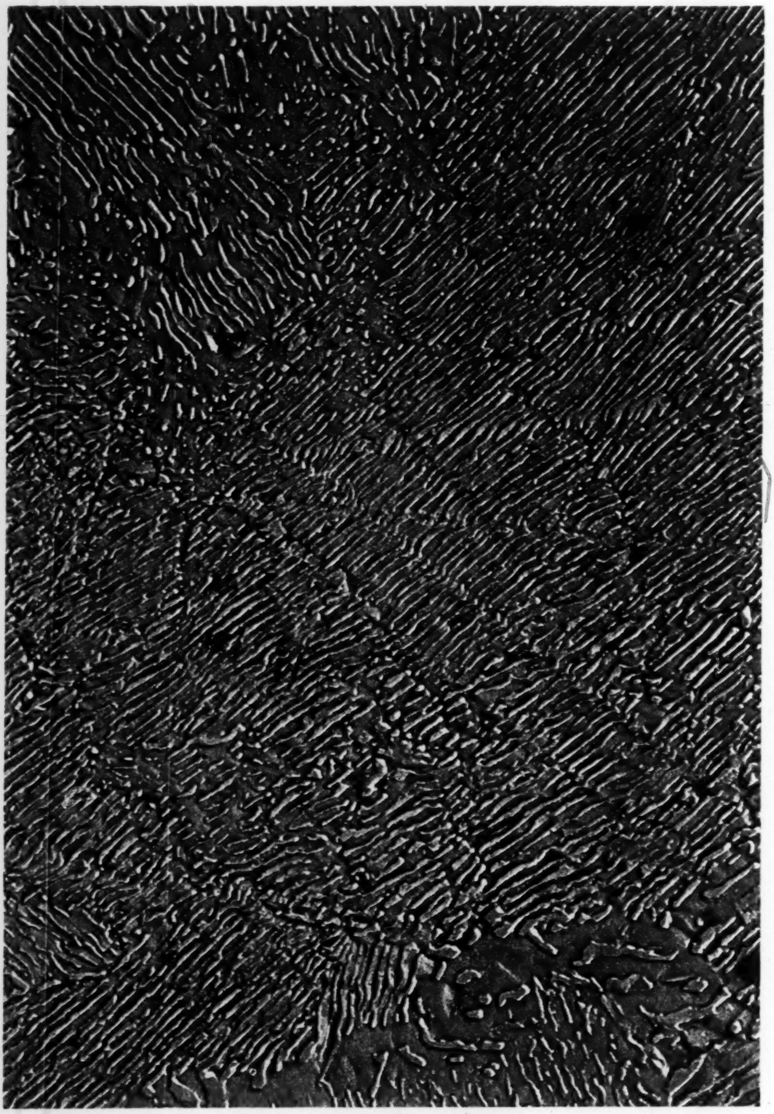


FIG. 11.—Electron Micrograph of Fine Pearlite, Isothermal Transformation at 1100 F., $FeCl_3$-HCl Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

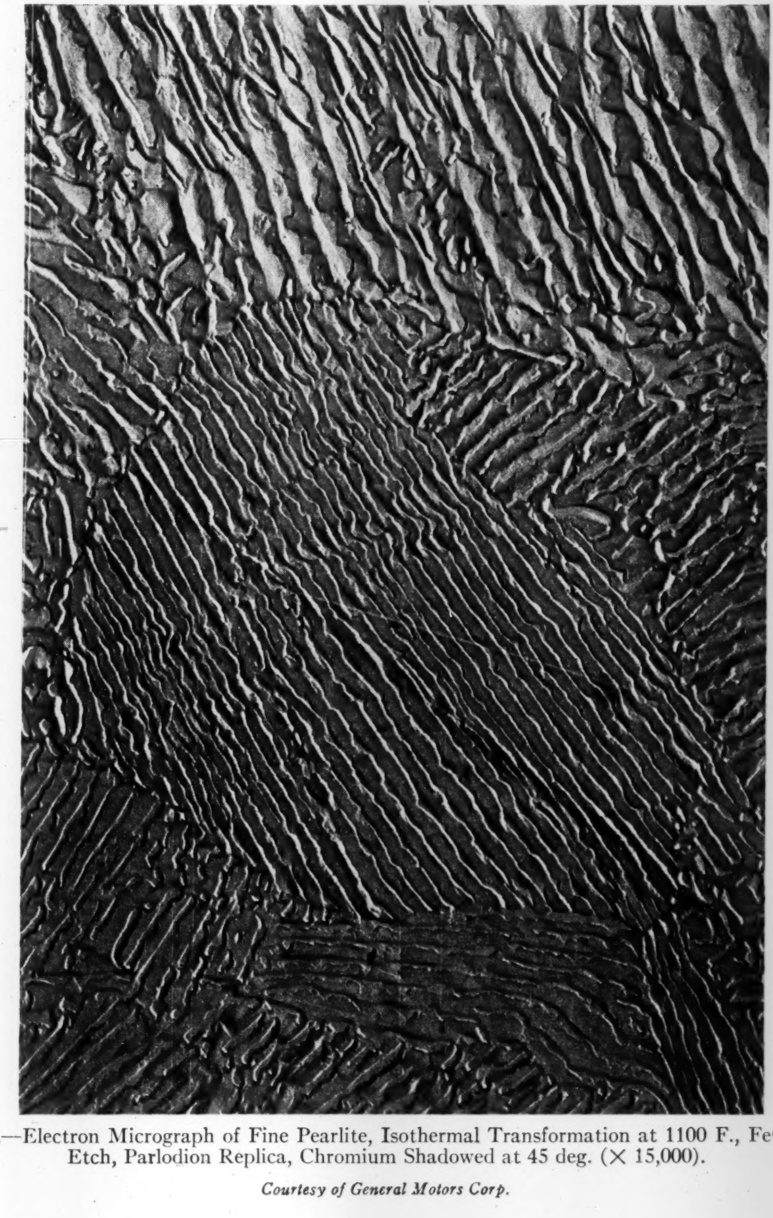


FIG. 12.—Electron Micrograph of Fine Pearlite, Isothermal Transformation at 1100 F., $FeCl_3$-HCl Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

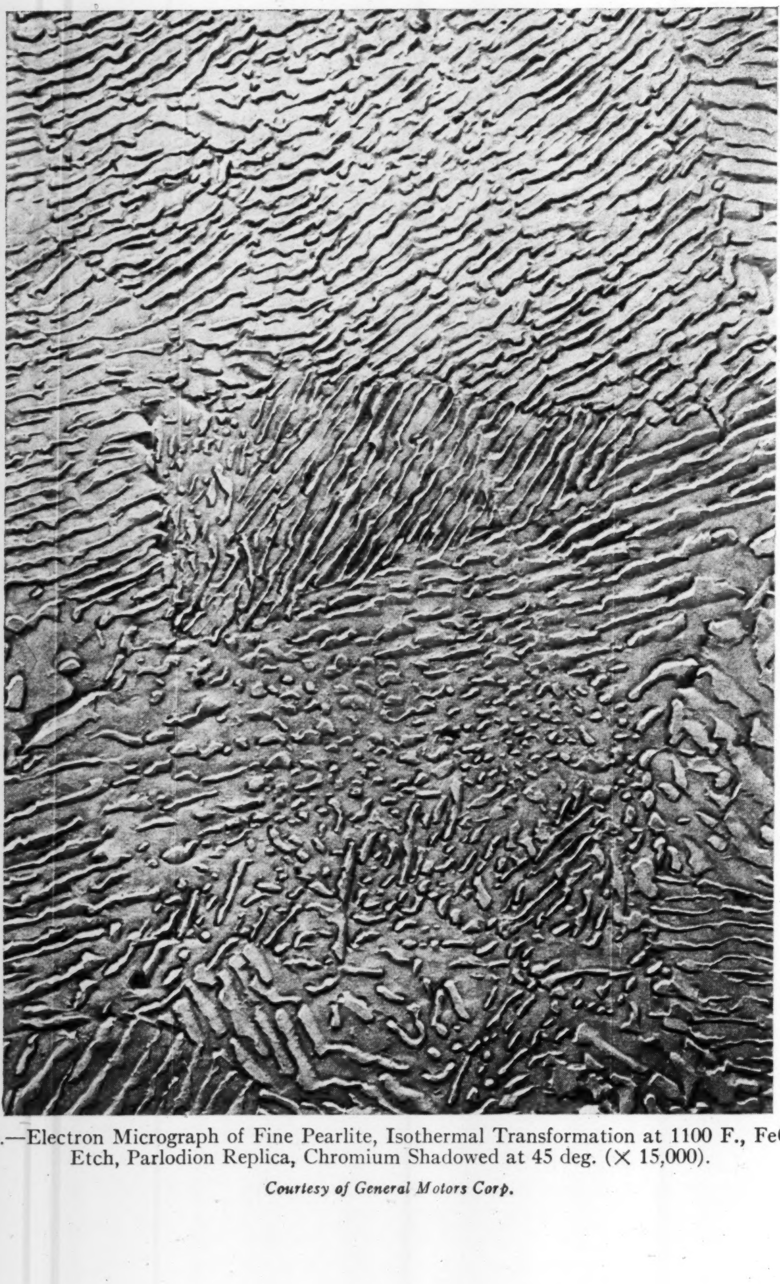
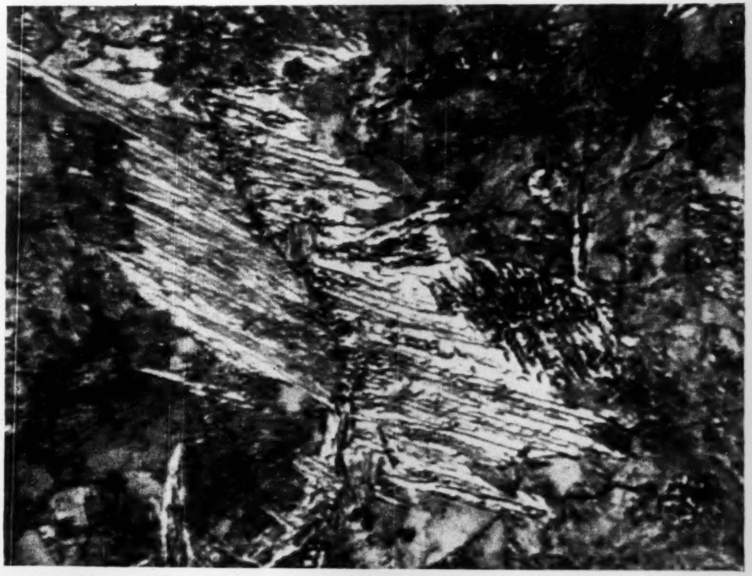


FIG. 13.—Electron Micrograph of Fine Pearlite, Isothermal Transformation at 1100 F., $FeCl_3$-HCl Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 14.—Light Micrograph of Fine Pearlite and Bainite, Isothermal Transformation at 950 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



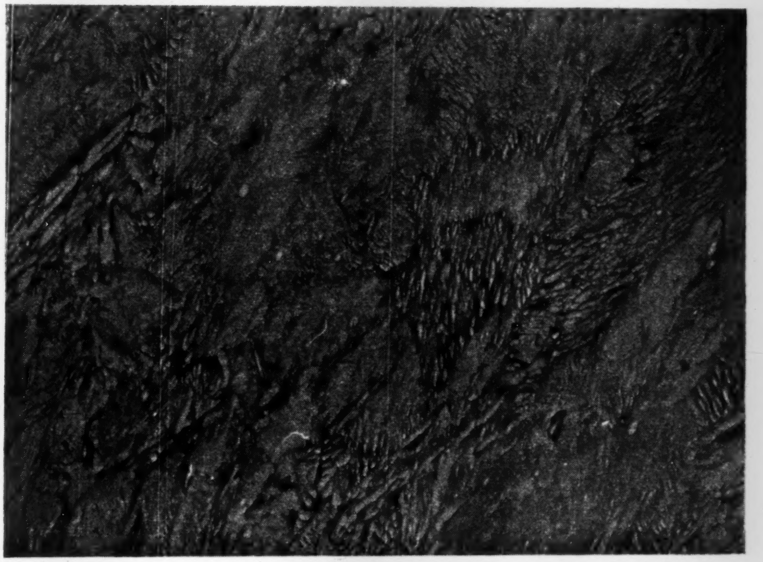


FIG. 15.—Electron Micrograph of Fine Pearlite and Bainite, Isothermal Transformation at 950 F., $FeCl_3$-HCl Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

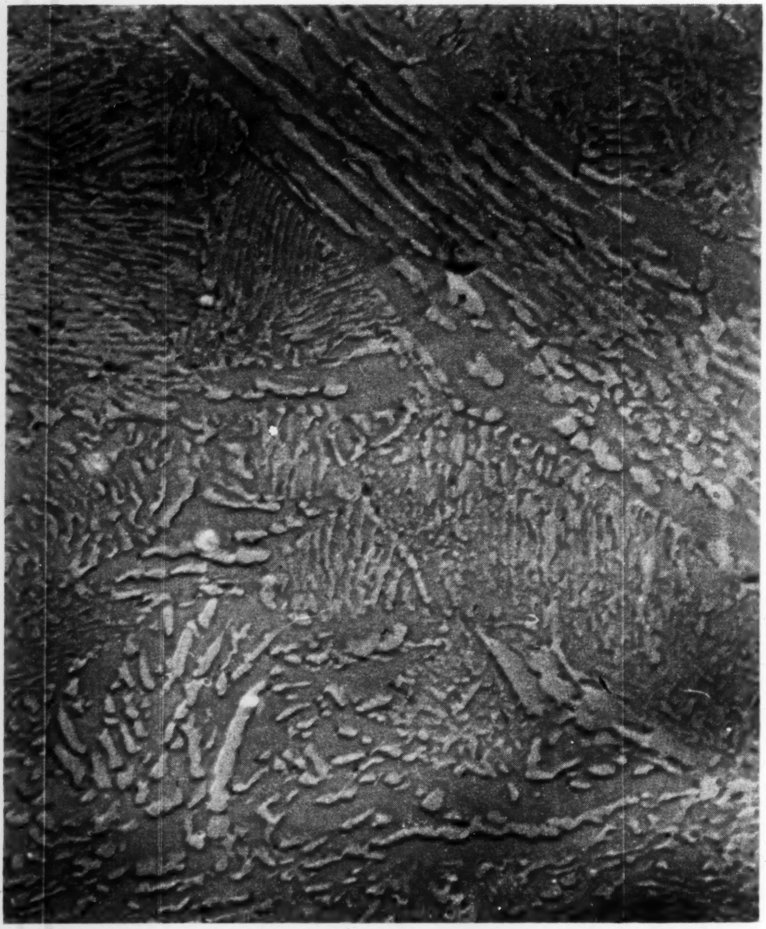


FIG. 16.—Electron Micrograph of Fine Pearlite and Bainite, Isothermal Transformation at 950 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of Chrysler Corp.

FIG. 17.—Light Micrograph of Bainite, Isothermal Transformation at 750 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.

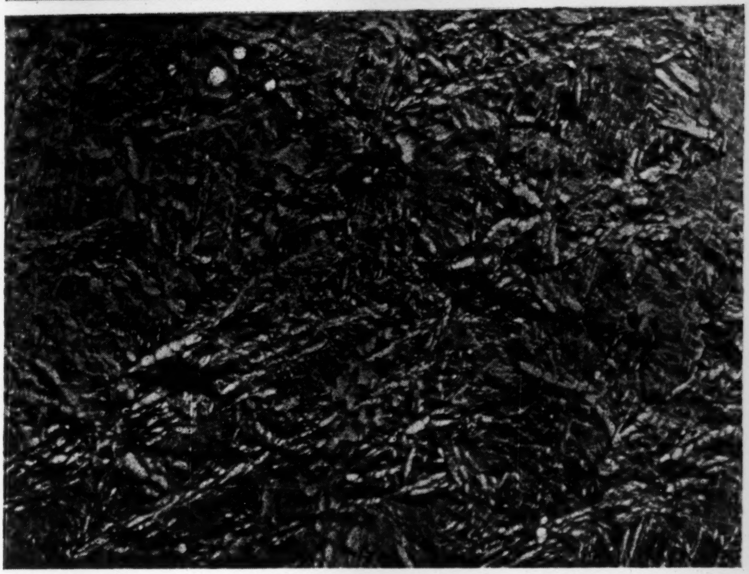


FIG. 18.—Electron Micrograph of Bainite, Isothermal Transformation at 750 F., $FeCl_3$-HCl Etch, Formvar Replica, Unshadowed (× 2500).

Courtesy of Chrysler Corp.

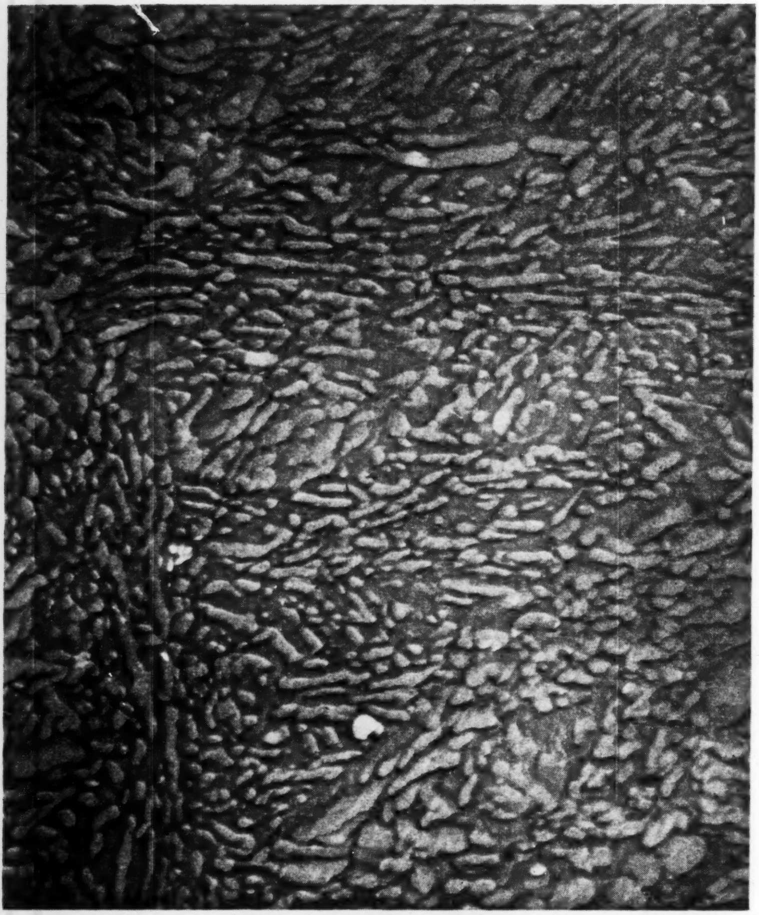
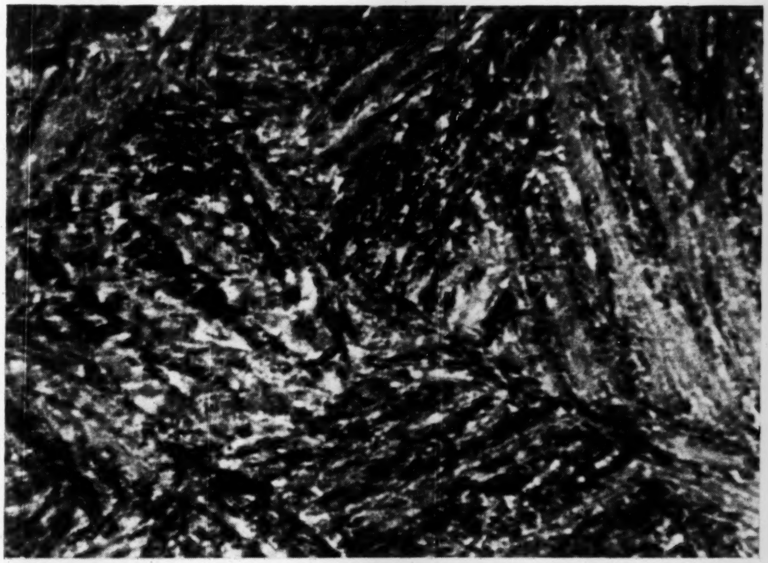


FIG. 19.—Electron Micrograph of Bainite, Isothermal Transformation at 750 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of Chrysler Corp.

FIG. 20.—Light Micrograph of Bainite, Isothermal Transformation at 500 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



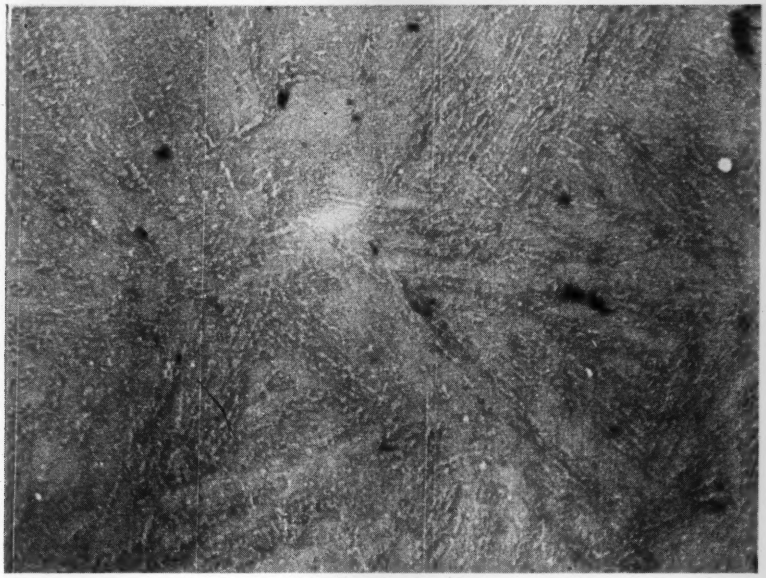


FIG. 21.—Electron Micrograph of Bainite, Isothermal Transformation at 500 F., Picral Etch Formvar Replica, Chromium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

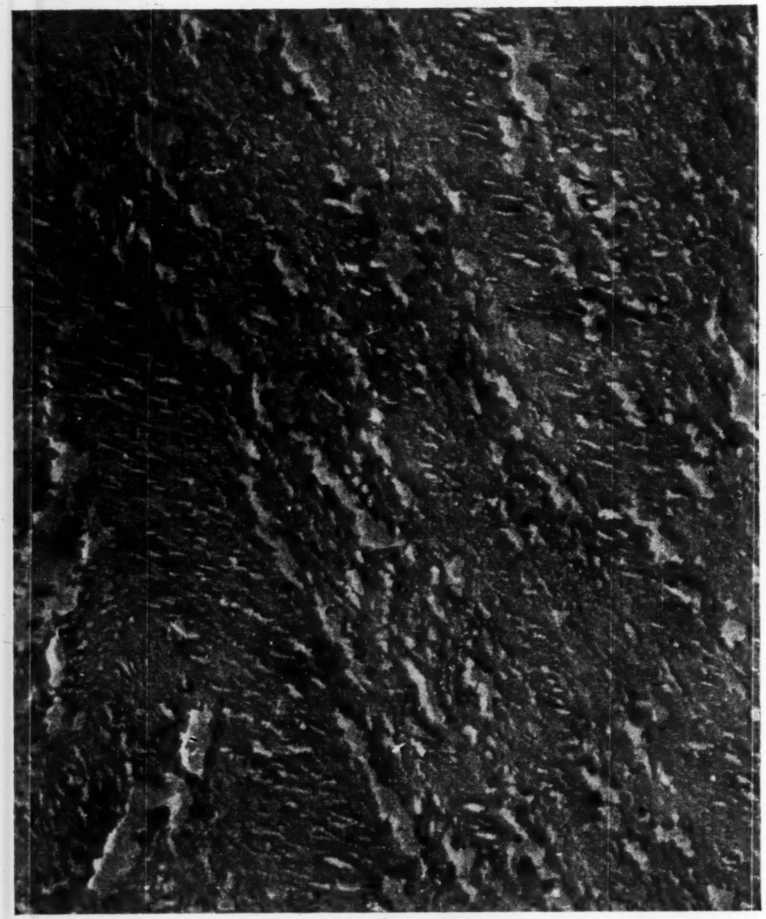


FIG. 22.—Electron Micrograph of Bainite, Isothermal Transformation at 500 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of Chrysler Corp.

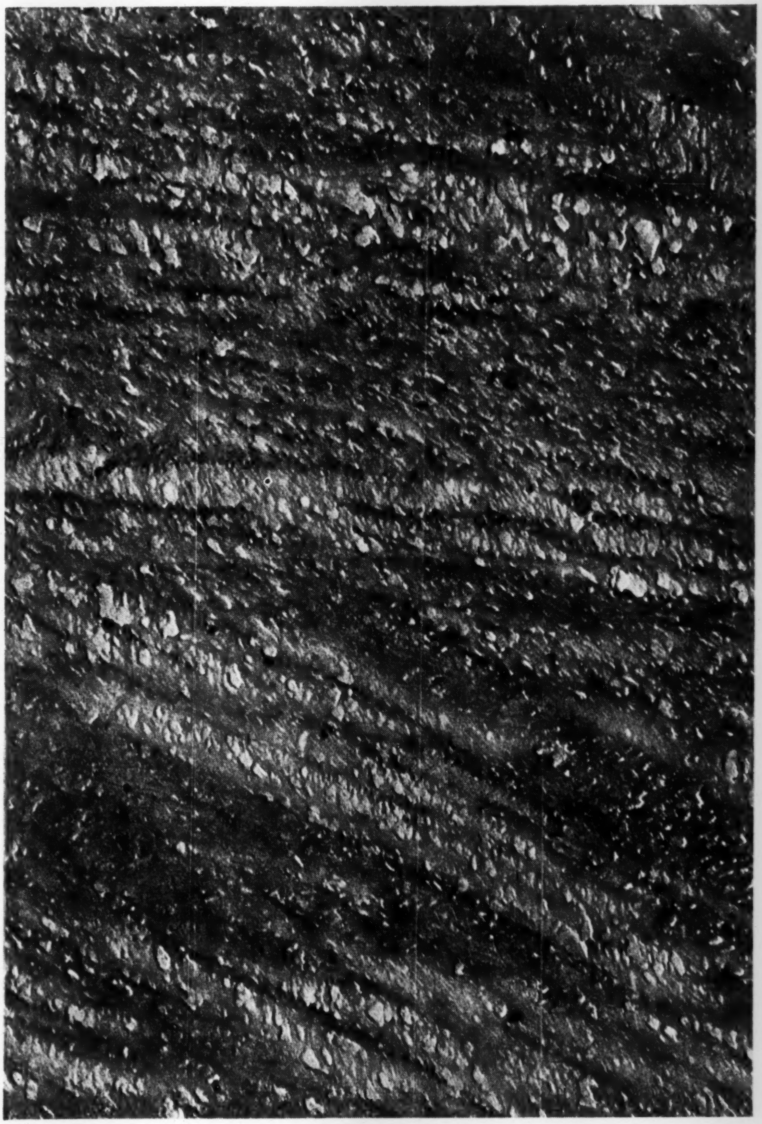


FIG. 23.—Electron Micrograph of Bainite, Isothermal Transformation at 500 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

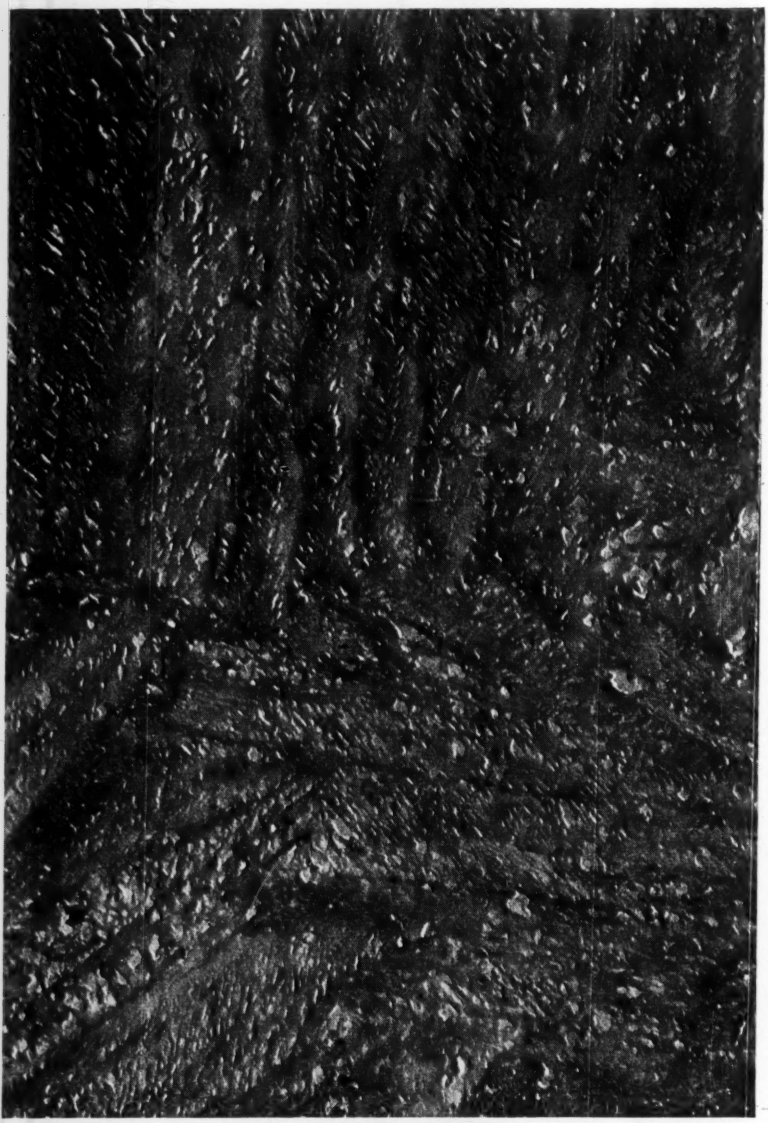
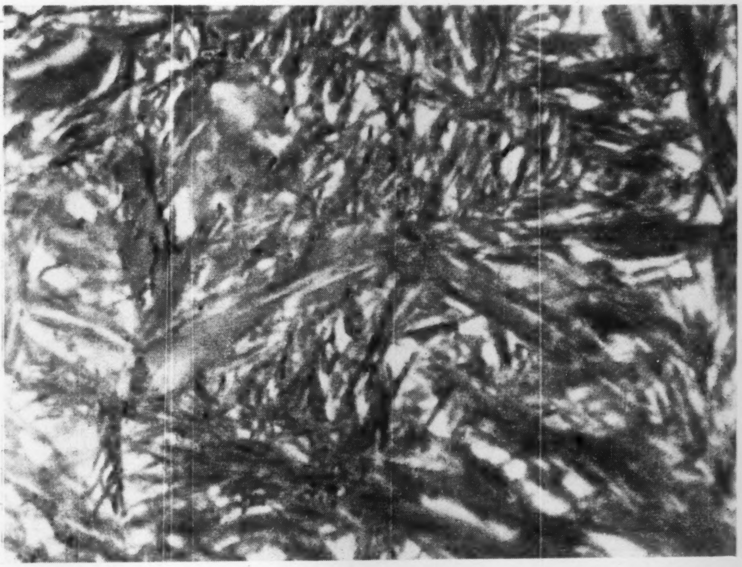


FIG. 24.—Electron Micrograph of Bainite, Isothermal Transformation at 500 F., Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 25.—Light Micrograph of Martensite, Brine Quenched, Picral Etch (× 2500).

Courtesy of United States Steel Corp.



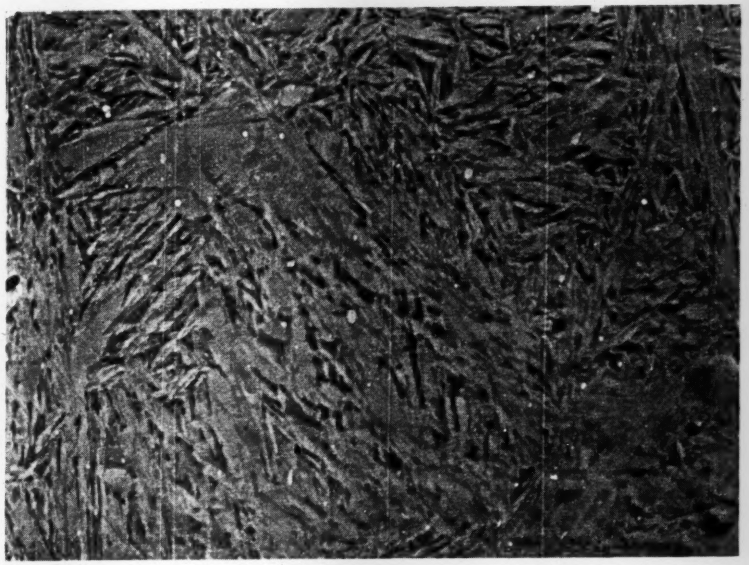


FIG. 26.—Electron Micrograph of Martensite, Brine Quenched, Picral Etch, Formvar Replica, Palladium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

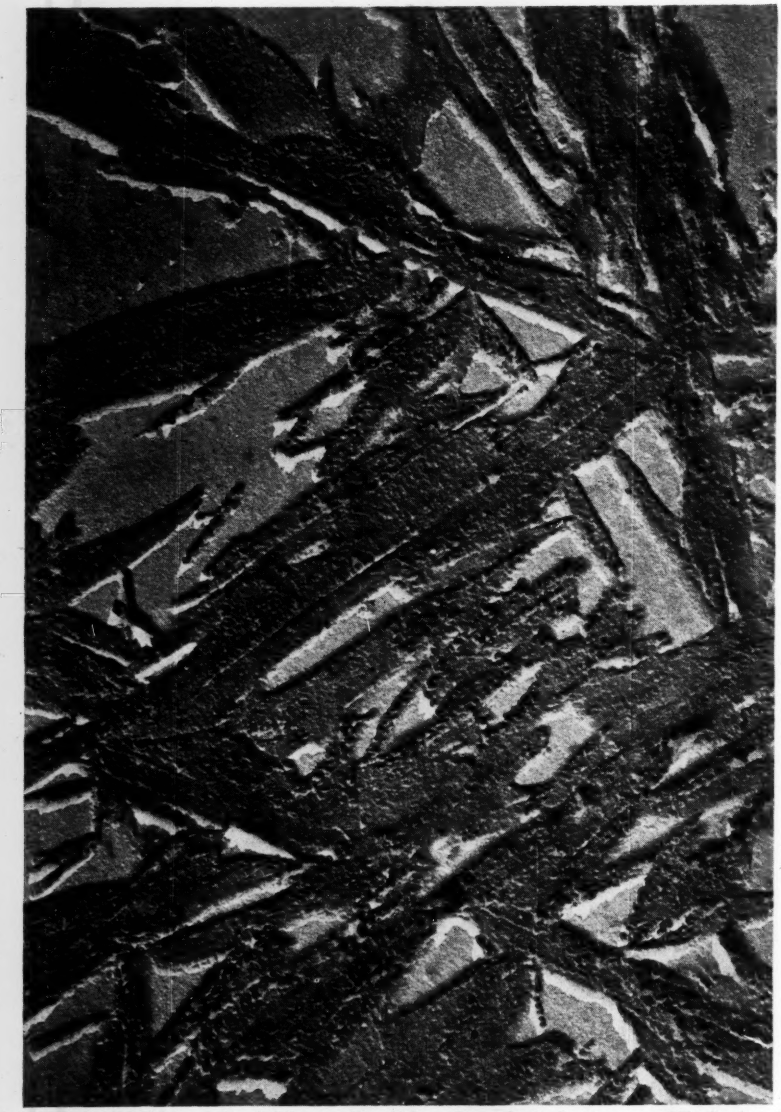
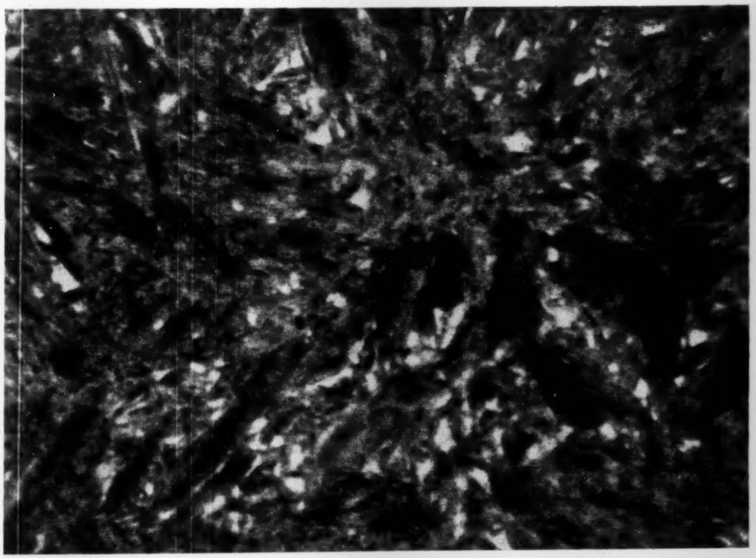


FIG. 27.—Electron Micrograph of Martensite, Brine Quenched, 2 per cent Nital plus Zephiran Chloride Etch, Formvar Replica, Chromium Shadowed at 27 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 28.—Light Micrograph of Tempered Martensite, 1 hr. at 400 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp



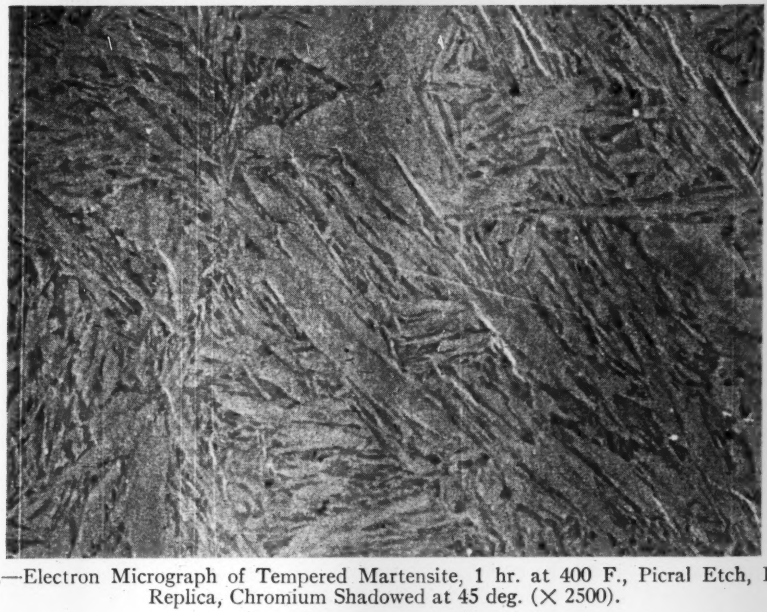


FIG. 29.—Electron Micrograph of Tempered Martensite, 1 hr. at 400 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

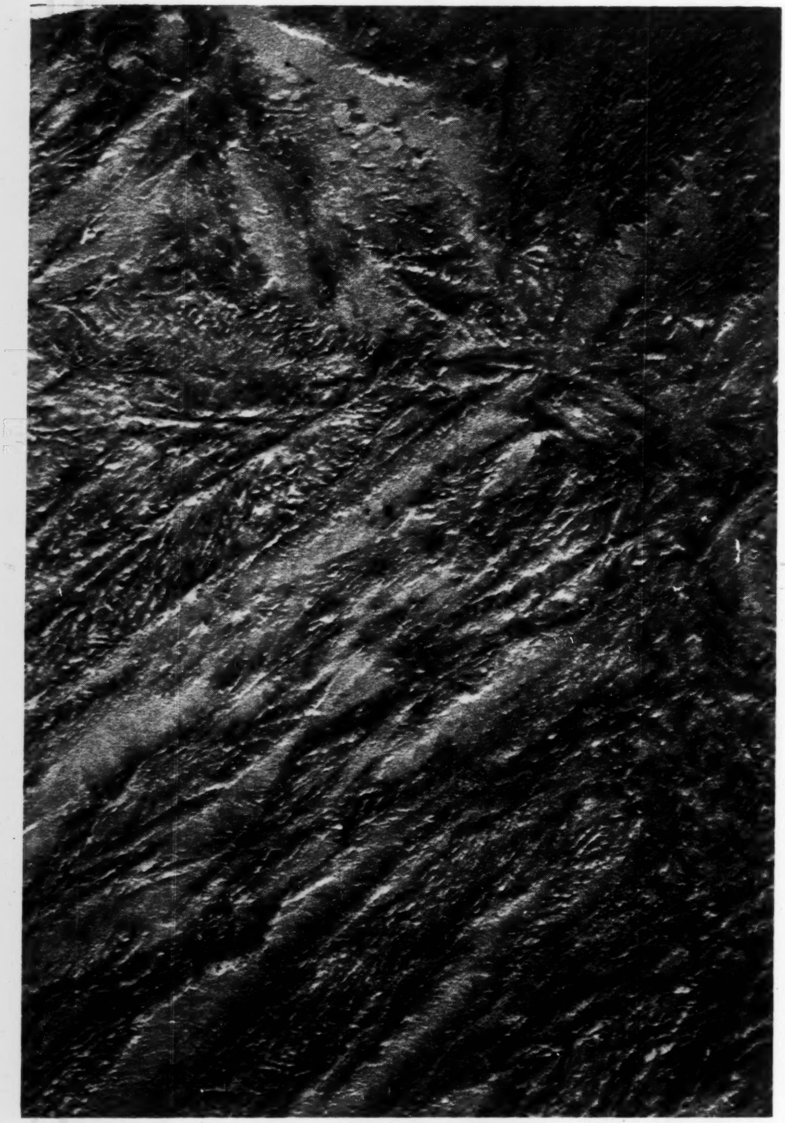
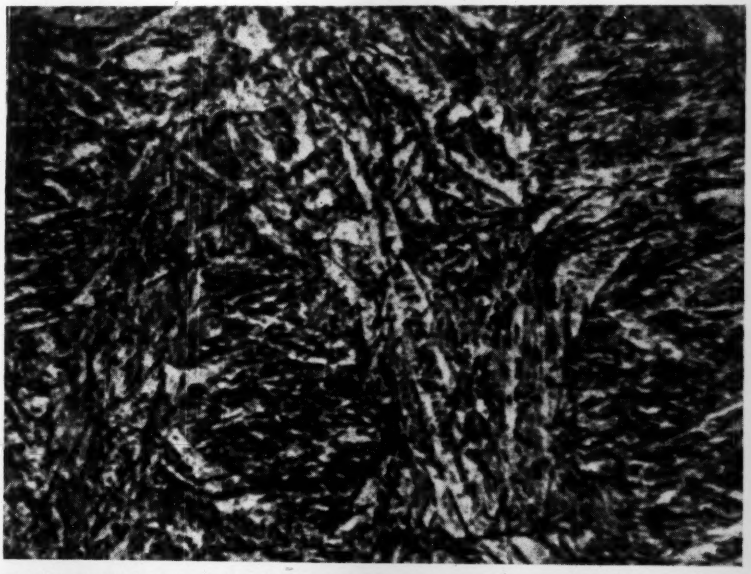


FIG. 30.—Electron Micrograph of Tempered Martensite, 1 hr. at 400 F., Picral plus Zephiran Chloride Etch, Formvar Replica, Chromium Shadowed at 27 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 31.—Light Micrograph of Tempered Martensite, 1 hr. at 600 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



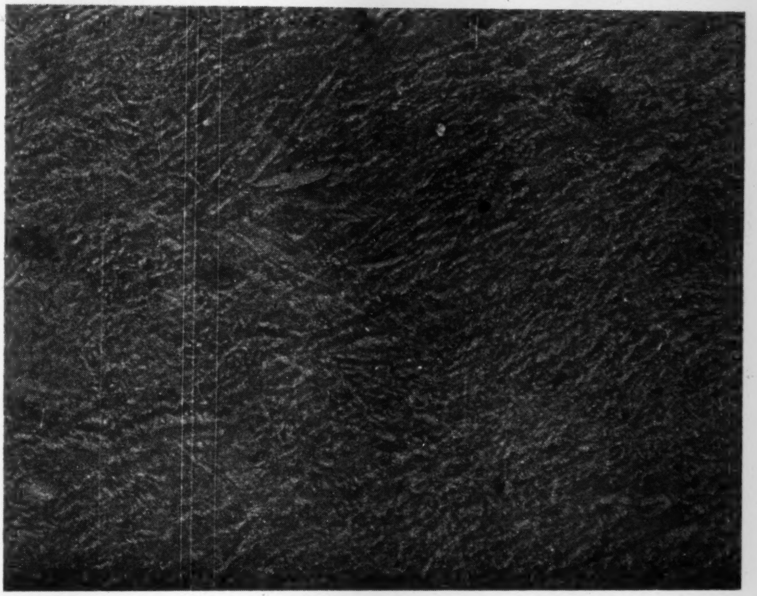


FIG. 32.—Electron Micrograph of Tempered Martensite, 1 hr. at 600 F., $FeCl_3$-HCl Etch, Formvar Replica, Unshadowed (× 2500).

Courtesy of Chrysler Corp.

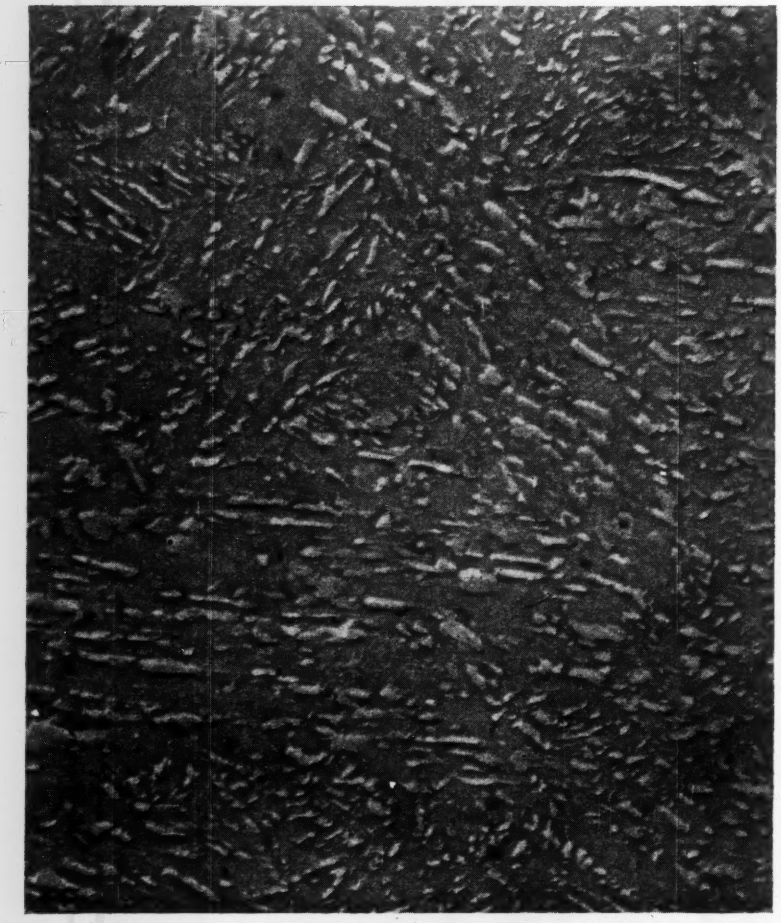
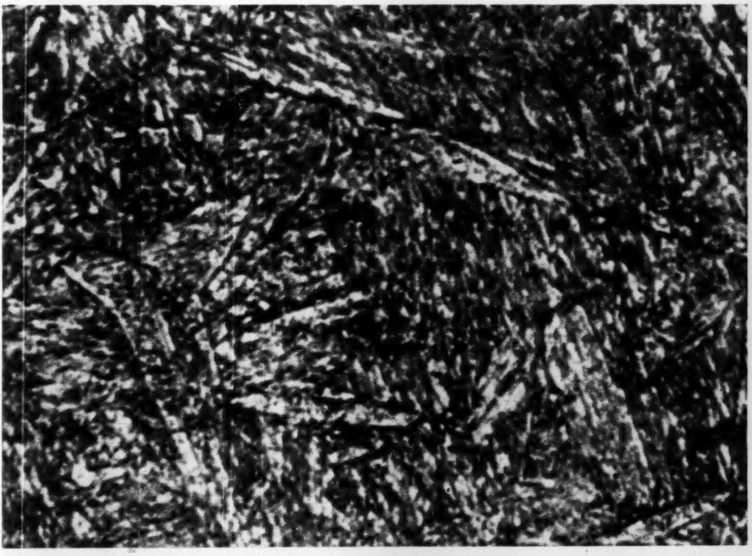


FIG. 33.—Electron Micrograph of Tempered Martensite, 1 hr. at 600 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of Chrysler Corp.

FIG. 34.—Light Micrograph of Tempered Martensite, 1 hr. at 800 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



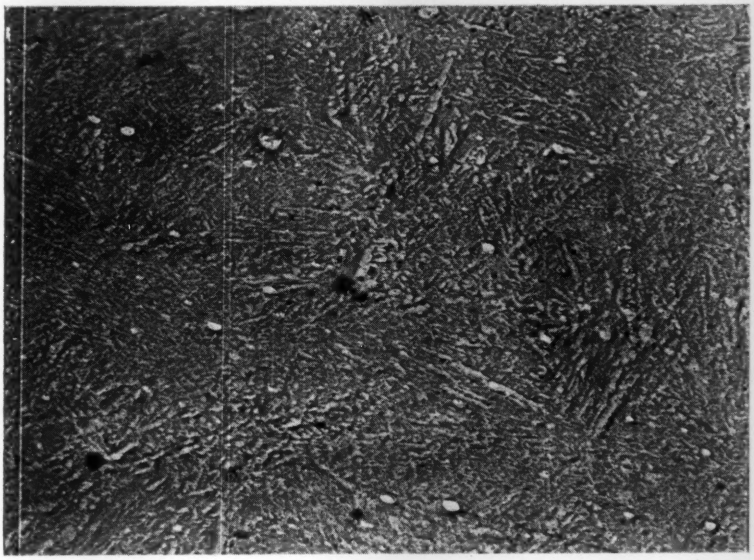


FIG. 35.—Electron Micrograph of Tempered Martensite, 1 hr. at 800 F., FeCl$_3$-HCl Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

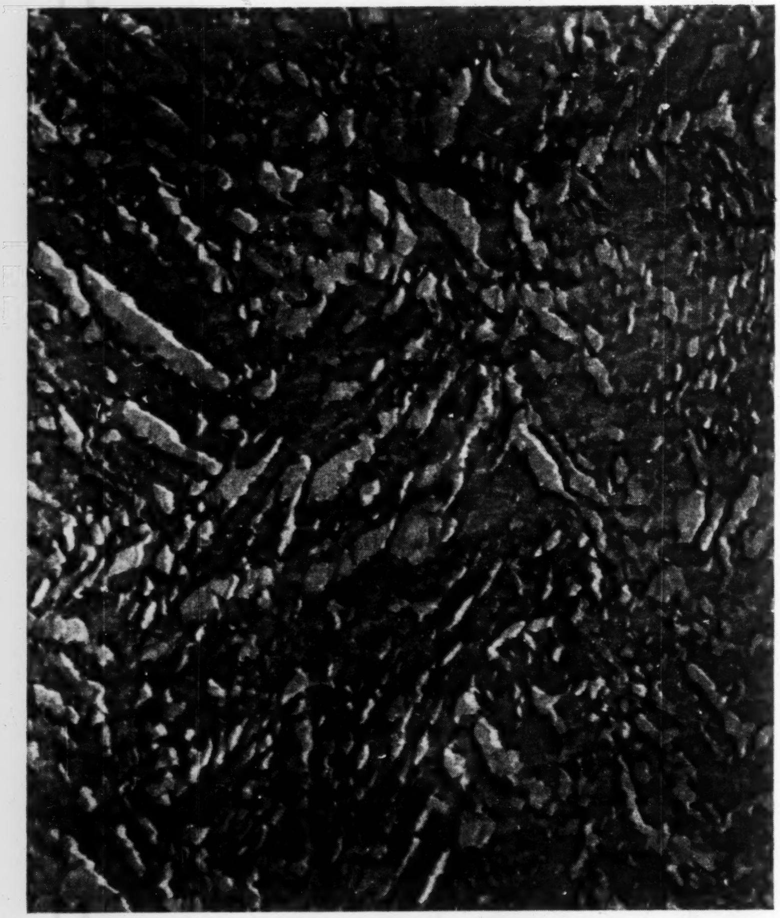


FIG. 36.—Electron Micrograph of Tempered Martensite, 1 hr. at 800 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of Chrysler Corp.

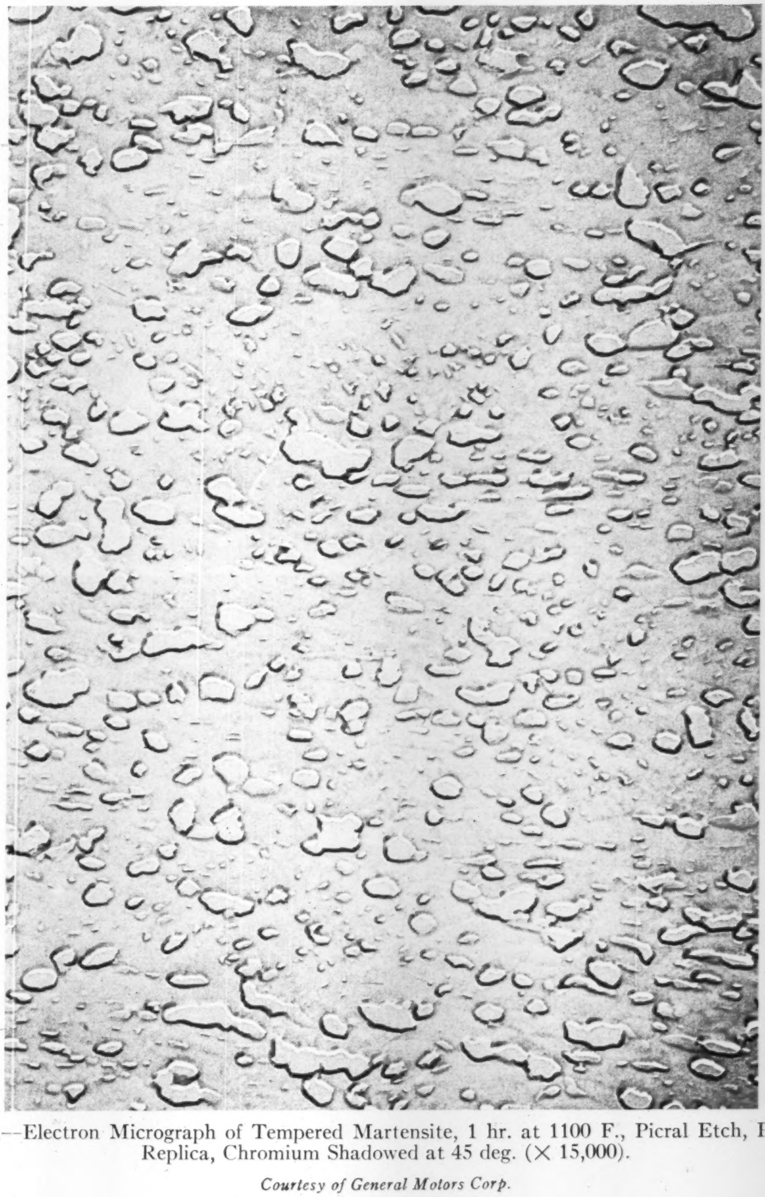
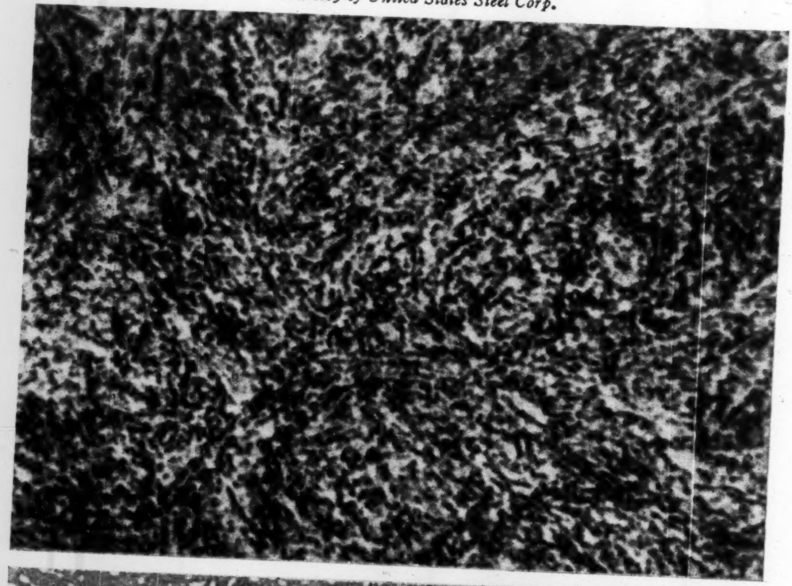


FIG. 39.—Electron Micrograph of Tempered Martensite, 1 hr. at 1100 F., Picral Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 37.—Light Micrograph of Tempered Martensite, 1 hr. at 1100 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



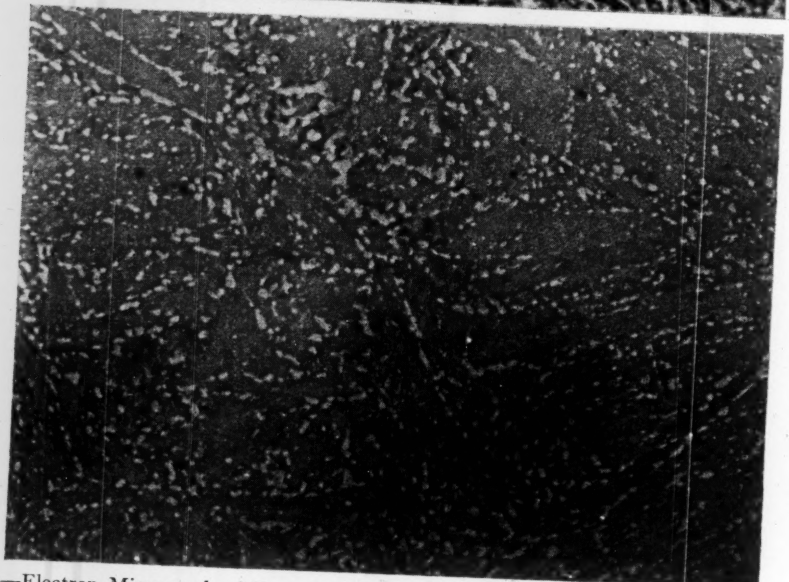
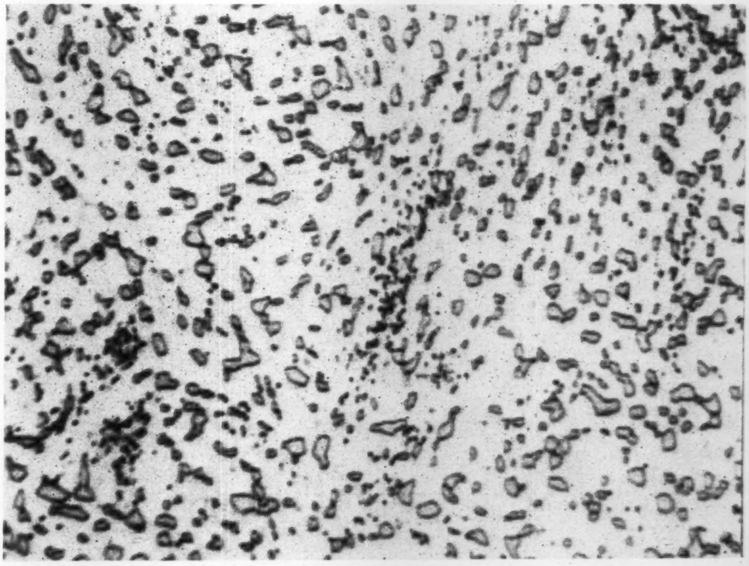


FIG. 38.—Electron Micrograph of Tempered Martensite, 1 hr. at 1100 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

FIG. 40.—Light Micrograph of Spheroidized Carbides, 12 hr. at 1250 F., Picral Etch (× 2500).

Courtesy of United States Steel Corp.



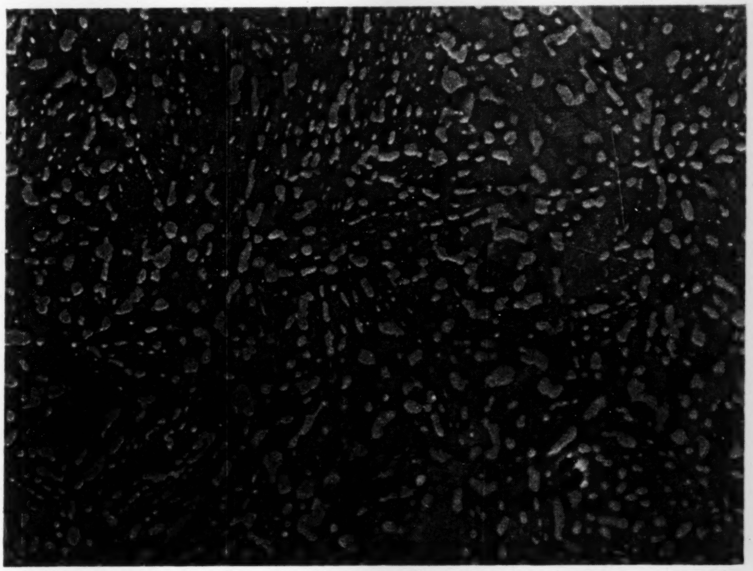


FIG. 41.—Electron Micrograph of Spheroidized Carbides, 12 hr. at 1250 F., Picral Etch, Formvar Replica, Chromium Shadowed at 45 deg. (× 2500).

Courtesy of Chrysler Corp.

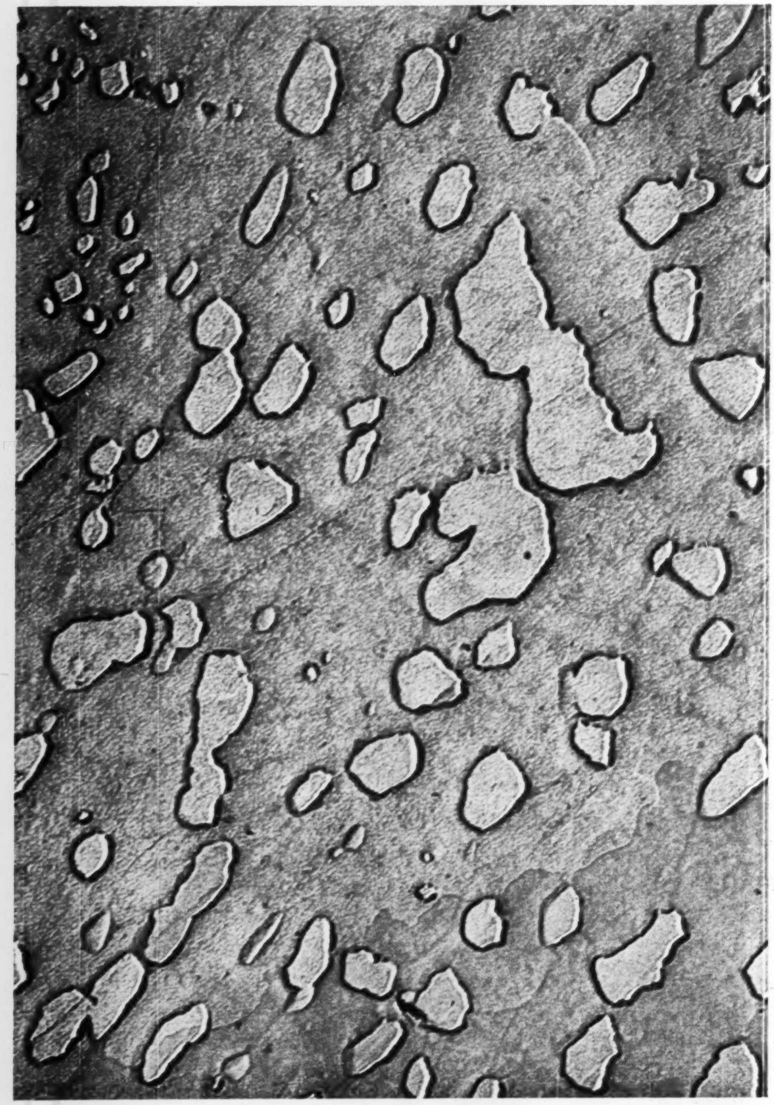
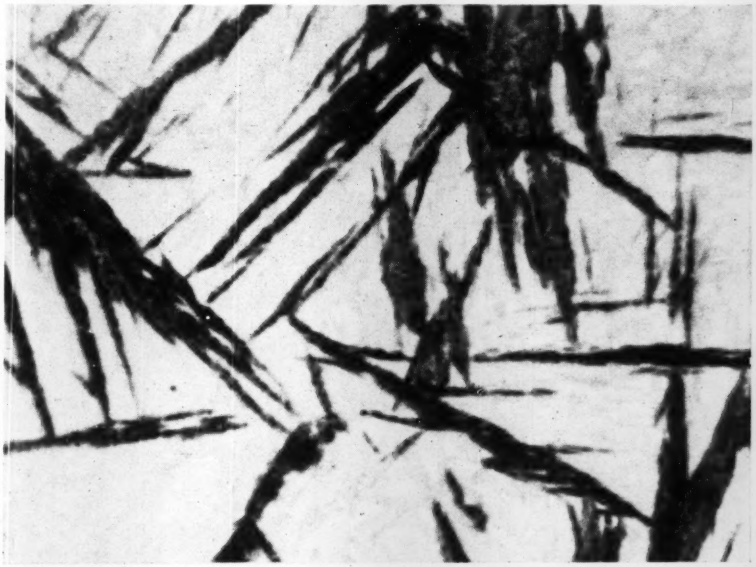


FIG. 42.—Electron Micrograph of Spheroidized Carbides, 12 hr. at 1250 F., Picral Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

FIG. 43.—Light Micrograph of Bainite, Partially Transformed at 500 F., Picral Etch (× 2500)

Courtesy of United States Steel Corp.



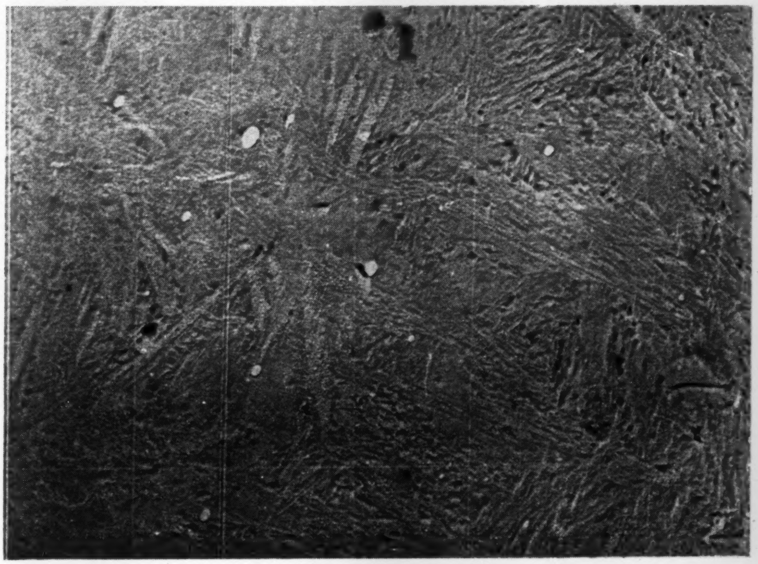


FIG. 44.—Electron Micrograph of Bainite, Partially Transformed at 500 F., Picral Etch, Formvar Replica, Palladium Shadowed at 27 deg. (× 2500).

Courtesy of Chrysler Corp.

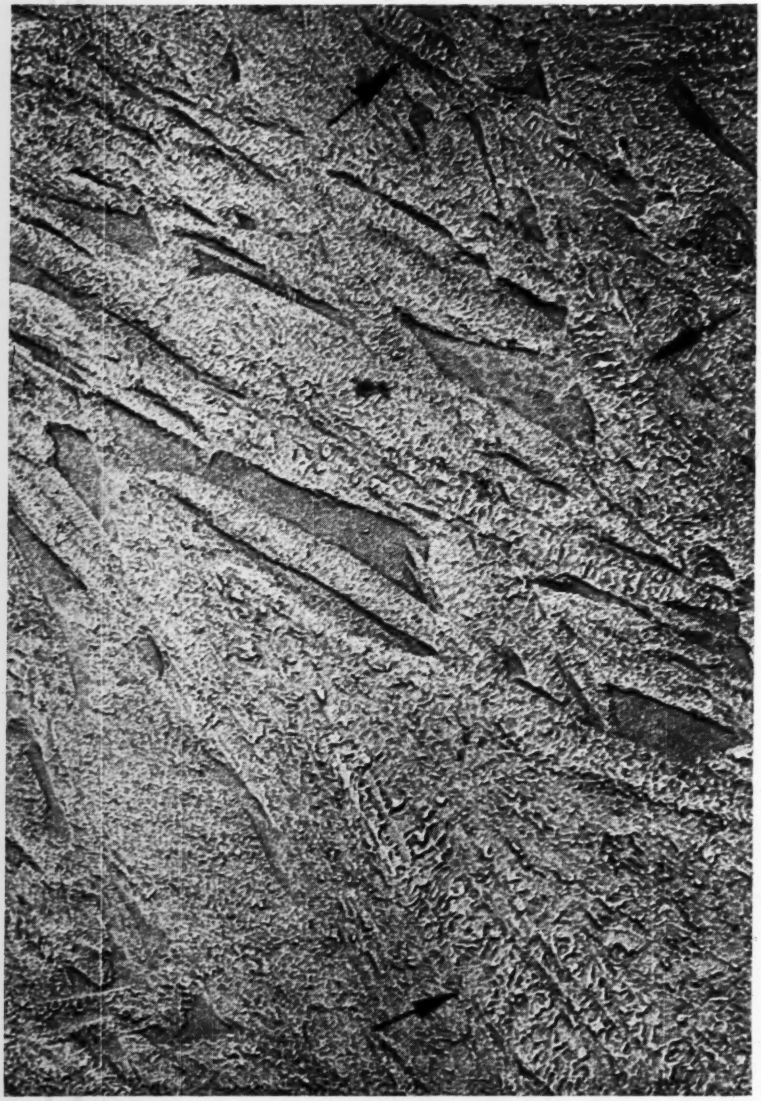


FIG. 45.—Electron Micrograph of Bainite, Partially Transformed at 500 F. (Arrows), Picral Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

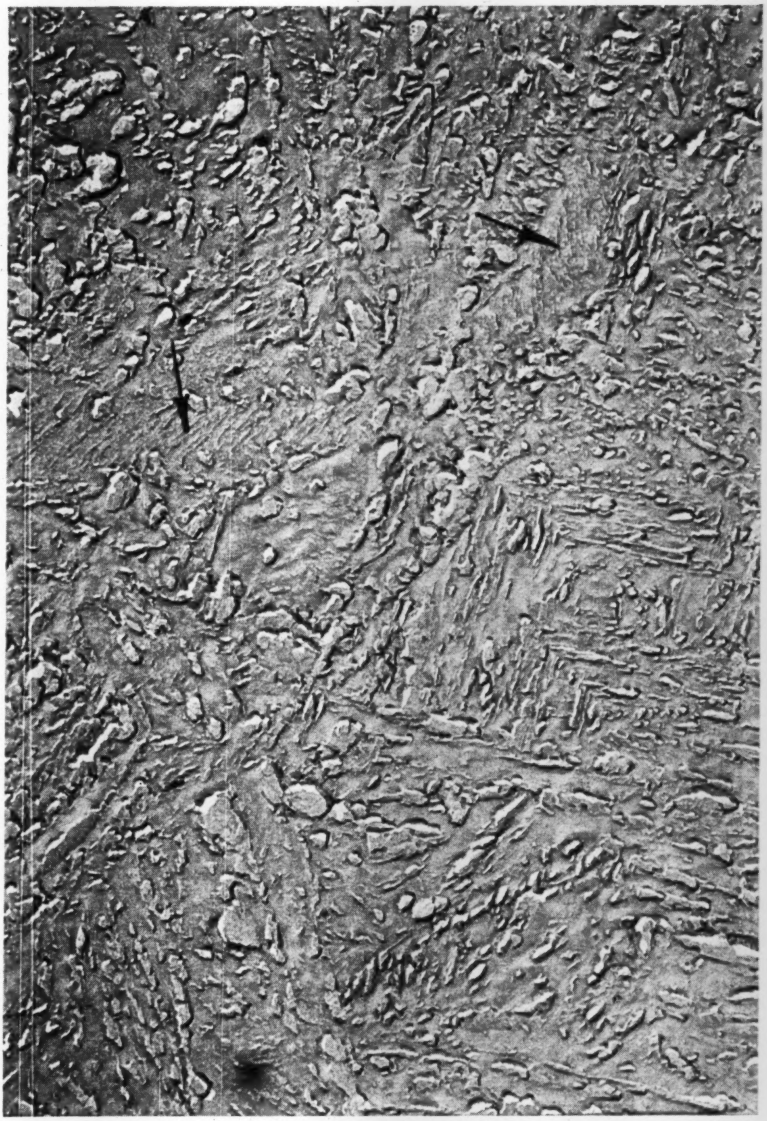


FIG. 46.—Electron Micrograph of Tempered Martensite, 1 hr. at 600 F., Picral Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

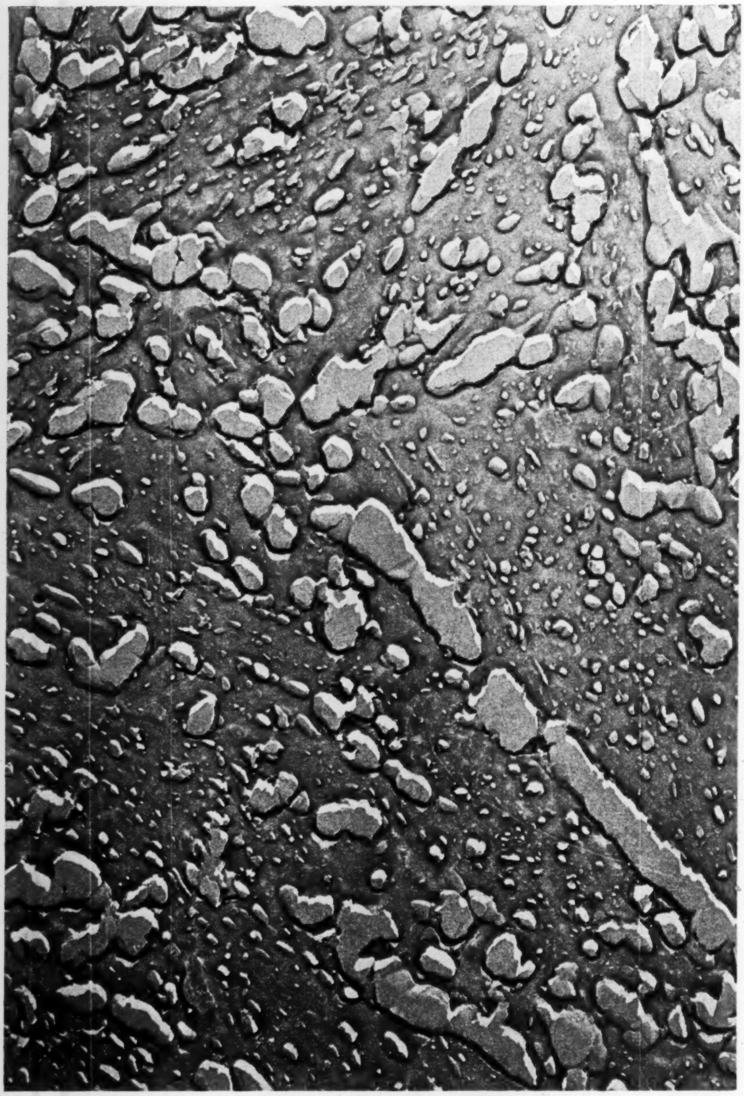


FIG. 47.—Electron Micrograph of Tempered Martensite, 1 hr. at 1100 F., Picral Etch, Parlodion Replica, Chromium Shadowed at 45 deg. (× 15,000).

Courtesy of General Motors Corp.

APPENDIX

SUMMARY OF TECHNIQUES EMPLOYED IN THE ELECTRON MICROSCOPE STUDY OF STEEL

Following is a brief summary of the methods which the members of the committee have applied to the study of steel microstructure with the electron microscope. Each step in the preparation of the final electron micrograph is listed, and the variations in procedure are indicated, together with the respective advantages and disadvantages, as judged by experience. The currently preferred technique is discussed first among the variations in each step of preparation.

It should be pointed out that the currently preferred techniques do not necessarily represent the best available, but are those which have been useful in the present investigation. In many instances active work is being continued on the application of other methods, or on the development of new methods, which will extend the available information.

I. SAMPLE PREPARATION

(*a*) *Mechanical Polishing:**

Technique.—The majority of specimens have been polished for electron microscope observation by mechanical procedures. These are essentially the same as those used for light-optical study. Alternate polishing and etching should always be employed to remove disturbed metal from the surface.

Advantages.—Utilizes standard techniques and equipment. Easier to reproduce results than with electropolishing.

Disadvantages.—May produce "worked" surface; some tearing at the edges of carbides and background roughness.

(*b*) *Electropolishing:*

Technique.—A minor number of laboratories have used electropolishing for the preparation of some specimens. The solutions which have been used are:

1. (Original Jacquet electrolyte).
   Bath.—1 part perchloric acid to 7 parts acetic anhydride, plus 50 ml. water per liter.
   Cathode.—Aluminum, about 50 v. d-c.
   Distance.—30 mm. with stirring.
2. (New Jacquet electrolyte, modified).
   Bath.—1 part perchloric acid to 6 parts acetic acid, plus 10 ml. $FeCl_3$-HCl etchant (see II (*b*)).
   Other conditions as above.
3. *Bath.*—50 ml. perchloric acid per liter of acetic acid or alcohol.

Advantages.—Produces surface free from "worked" metal.

Disadvantages.—Electropolishing equipment and techniques are less commonly available, although the equipment may be easily constructed. There are some hazards in handling the preferred electrolytes, and difficulty in reproducing results.

II. ETCHANTS

(*a*) *Picral:**

Technique.—The specimen is etched in a solution containing 4 per cent picric acid in 95 per cent ethyl alcohol. The solution is commonly activated with Fe^{+++} ion, or by standing some time in contact with iron metal.[1]

Etching time is 5 to 90 sec.; less than 30 sec. is considered a light etch, over 30 sec. is a heavy etch. Light etches are recommended for fine pearlite and the bainites, although some laboratories prefer a light etch for all structures.

After etching, the sample is washed according to either Sequence A or Sequence B as follows:

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* Currently preferred technique.

[1] As in light-optical metallography, zephiran chloride is sometimes used to activate picral.

Sequence A

1. 50 per cent methyl alcohol—50 per cent acetone mixture, plus ½ per cent citric acid to minimize surface oxide formation.
2. 50 per cent methyl alcohol—50 per cent acetone (second, clean wash).
3. C.P. benzene.
4. Dry with warm air.

Sequence B

1. Wash in hot running water.
2. Flood with liquid soap and scrub with finger (or cotton swab).
3. Rinse under hot running water (or alcohol).
4. Blow dry.

Protect the surface immediately with a coat of formvar in chloroform.

Advantages.—Distinguishes the various steel structures more satisfactorily. It is a standard etchant, widely used in light-optical metallography. Replicas may strip more readily from a surface etched with picral.

Disadvantages.—Does not differentiate between the earlier (below 700 F.) progressive stages in the tempering of martensite as well as ferric chloride. The same objection may hold true for bainite. Etch products may be formed that strip with the replica and confuse interpretation, especially with the pearlites and upper bainite.

(*b*) *Ferric Chloride–Hydrochloric Acid:*

Technique.—The etchant contains:

1 per cent HCl
2 per cent $FeCl_3$
Balance, 95 per cent ethyl alcohol, or methyl alcohol

The procedure is similar to that outlined above, excepting that the etching times are slightly shorter.

Advantages.—May form less etch-products and oxide on surface; differentiates carbide gradients and crystal structure better.

Disadvantages.—Etching action has been less extensively studied by metallurgists. Replicas are more difficult to strip from $FeCl_3$-HCl etched surface.

(*c*) *Nital plus Zephiran Chloride:*

Technique.—The etchant contains:

50 ml. 2 per cent nital
5 to 6 drops zephiran chloride (concentrated)

Etching times are generally much shorter than those of picral or $FeCl_3$-HCl.

Advantages.—Most satisfactory for etching as-quenched and slightly tempered martensitic structures.

Disadvantages.—Exceedingly active and difficult to control. Over-etches pearlite and bainite structures.

(*d*) *Electrolytic Etching:*

Technique.—The specimen is made the anode in an electrolyte. One solution which has been used is the $FeCl_3$-HCl etchant.

Electrolytic etching methods have been used to only a limited extent. However, present indications are that it is well suited for developing microstructures to be examined electron-optically, provided close control of variables, such as electrolyte, temperature, voltage, etc. is exercised.

(*e*) *Gas Ion Discharge:*

Technique.—The specimen is made the cathode in a vacuum chamber containing argon or air at a pressure of 15 to 30 μ of mercury. A d-c. potential (a-c. may be used) of 5000 to 10,000 v. is applied to cause a current of 5 to 10 ma. to flow.

Advantages.—Produces a very clean surface and provides a means of etching microstructures that are etched only with difficulty by other means.

Disadvantages. — Requires specialized equipment and etching times are long. (The etching time is greatly reduced by placing the plane of the surface to be etched at right angles to the anode.) Sufficient results using this technique have not yet been obtained to allow complete evaluation.

III. REPLICAS

(*a*) *One-Step Plastic Replicas:**

Technique.—Etched surface is covered with solution of plastic, draining off excess.

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* Currently preferred technique.

Several solutions are in general use, including the following:

1. Fresh,[2] 0.75 per cent solution of formvar in freshly distilled dioxane.[3] Replica is removed by moistening surface with breath, and stripped with scotch tape upon which a screen has been placed.
2. Fresh 0.5 per cent solution of formvar in dioxane. Replica is removed by stripping under warm water. Formvar replicas stripped by this method show no strain marks.
3. 0.75 per cent solution of parlodion in amyl acetate. Replica is removed by any of several methods, including the use of a heavy polyvinyl alcohol backing film. Parlodion films appear to reproduce fine detail more satisfactorily, and are less likely to show holes and strain marks.
4. 2 per cent ethyl cellulose in ethylene dichloride.

Shadowing of the Replica.—This step is essential for good contrast and detail.

Advantages.—Rapidity and simplicity of equipment. The resulting electron micrographs show the same relief perspective as the original specimen surface, providing a high shadow angle and direct-printing is used. (See discussion of shadowing and photographic technique). These micrographs are more readily interpreted by metallurgists with light-optical experience.

Disadvantages.—Only moderate contrast and detail is obtained; there is difficulty in stripping, particularly from heavily etched surfaces.

(*b*) *Polystyrene-Silica:*

Technique.—Polystyrene is molded against the etched steel surface at a pressure of 4000 to 8000 psi. and a temperature of 325 F. After cooling, the polystyrene is separated from the metal specimen and the plastic replica surface coated with SiO_2 by vacuum evaporation. (SiO or LiF may also be used, and are easier to evaporate.) The silica replica is stripped from the polystyrene in ethyl bromide containing 3 per cent benzene.

Shadowing.—Infrequent.

Advantages.—Ability to show very fine detail; extreme contrast.

Disadvantages.—Difficulty of interpretation and correlation with light-optical micrographs. Greater time and more elaborate equipment necessary for preparation. Polystyrene molding conditions will temper quenched martensite slightly.

(*c*) *Two-Step Positive Replicas:*

Technique.—The etched surface is covered with a meniscus of 15 per cent polyvinyl alcohol (low viscosity) in water. When dry the thick negative is stripped and the structure surface is replicated with formvar. The composite is placed formvar side up on water and the polyvinyl alcohol allowed to dissolve. The floating formvar positive is washed by drawing off the solution and replacing two or three times with clean warm water.

An alternate method uses either 8 per cent cellulose nitrate in amyl acetate or 10 per cent formvar in ethylene dichloride for the thick negative. After stripping, positive replicas are made with SiO or LiF. The negative is dissolved away by condensing vapors of a suitable solvent on the replica as it rests on a cooled specimen screen.

Shadowing.—Infrequent.

Advantages.—The thick negative replicas are easily stripped.

Disadvantages.—More time-consuming than direct stripped negative replicas. Electron micrographs of shadowed positive replicas must be reverse-printed in order to maintain the correct relief perspective of the original specimen. Artifacts are prevalent in polyvinyl alcohol negative replicas.

IV. SHADOWING OF REPLICAS*

Shadowing is necessary on plastic replicas to obtain contrast and to render the smaller detail visible.

Shadowing Materials.—Chromium, palladium, platinum, and uranium.

Equipment.—The shadowing materials are evaporated from a tungsten filament in a high vacuum chamber at pressures less than 10^{-4} mm. of mercury. The shadowing metal may be contained in a spiral basket or plated onto a hairpin filament.

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[2] Orientation of formvar develops after a few days in solution.

[3] ***Caution,*** peroxides, distillation from metallic sodium recommended.

* Currently preferred technique.

Shadowing Angle.—The shadowing angle varies from 1:1 (45 deg. to the plane of the replica) to 2:1 (27 deg. to the plane of the replica). The lower angle is preferable for fine structures, whereas the higher angle is used for coarse structures and yields better results with a direct print. Contrast is achieved with this high shadow angle technique through the formation of a "shadow cap."

V. PHOTOGRAPHY

Plates:

1. Contrast lantern-slide emulsion.*
2. Medium lantern-slide emulsion (The new Ilford emulsions appear promising)

Photographic Paper.—F-3, F-4, F-5.

Prints.—Direct print* for one-step replicas. Optional for polystyrene-silica and two-step replicas.

Reverse prints are made from a positive plate, contact-printed from the negative.

Magnification.—2500 × and 15,000 × on final prints.

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* Currently preferred technique.

REPORT OF COMMITTEE E-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION[1]

Committee E-5 on Fire Tests of Materials and Construction held one meeting during the past year in Pittsburgh, Pa., on March 1, 1950. Meetings of subcommittees preceded this meeting.

The committee now consists of 56 members, of whom 25 are classified as producers, 9 as consumers, 16 as general interest members, and 6 as consulting members.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, A. L. Brown.
Vice Chairman, W. J. Krefeld.
Secretary, H. M. Robinson.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Method of Fire Hazard Classification of Building Materials as appended hereto.[2] A standard for establishing the fire hazards of materials is urgently needed by manufacturers, building code authorities, and other interests. Several test methods have been used for this purpose, but only one produces data on all of the factors considered necessary for a proper classification of fire hazard. These factors are "ease of ignition," "rate of flame spread," "rate of combustion and heat generated." The committee's activities have so far been confined to establishing a test method for determining the fire hazards of wall and ceiling finish materials. The recommended tentative test method is, however, applicable to a wider range of materials and was developed by very careful study and research. Many materials were tested and the fire hazards of materials are now being established by this method at the Underwriters Laboratories, Inc. Further study and research now under way may result in a reduction in the size of equipment needed and the costs of making tests.

TENTATIVE REVISION OF STANDARD

Standard Methods of Fire Tests of Building Construction and Materials (**E 119 - 47**).[3]—The committee recommends the following additional new method of test, as appended hereto,[4] and the revision of Section 26 for publication as tentative revisions:

Alternate Test of Protection for Structural Steel Beams and Girders.—To be added as Sections 27 to 30 inclusive. This new alternate method permits conducting fire tests of structural floor beam and girders without application of load, the limits of fire endurance being defined by the temperatures of the steel at critical locations. The relation between strength and temperatures of floor beam and girders has been quite well established in full-scale tests under load.

Section 26(c).—Revise lines 5 and 6 to read: "more than 250 F. (139 C.) in the case of floors and 400 F. (222 C.) in the case of roofs, above the initial temperature."

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The new tentative method was accepted by the Society and appears in the 1950 Supplement to ASTM Book of Standards, Part 4.

[3] 1949 Book of A.S.T.M. Standards, Part 4.

[4] The new method was accepted as a "tentative revision" and appears in the 1950 Supplement to ASTM Book of Standards, Part 4.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

Standard Methods of Fire Tests of Building Construction and Materials (**E 119 - 47**).[3]—The committee recommends adoption as standard of the tentative revisions pertaining to Sections 3(*a*), 3(*b*) and 5(*b*).

Standard Method of Test for Combustible Properties of Treated Wood by the Crib Test Method (**E 160 - 46**).[2]—The committee recommends that the tentative revision of this standard, a new Section 4, be approved for reference to letter ballot of the Society for adoption as standard.

Standard Method of Tests for Combustible Properties of Treated Wood by the Fire Tube Test Method (**E 69 - 47**).[2]—The committee recommends that the new Section 4 be approved for reference to letter ballot of the Society for adoption as standard.

GENERAL ACTIVITIES OF THE COMMITTEE

Standard Specifications for Fire Tests of Materials and Construction.—Study is being given to the advisability of not only conducting fire tests on wood stud partitions loaded to the extent that full allowable stress will be developed in the studs, but also loaded only to the extent contemplated in actual use. The need for a more precise defining of procedure for conditioning specimens for fire testing is under consideration.

Fire Tests of Lumber.—The need of a standard method of test for evaluating fire-retardant paints is under discussion.

Fire Tests of Acoustical and Similar Finishes.—Two study groups have been appointed, one to investigate the possibility of simplifying and reducing the cost of performing tests on interior wall and ceiling finish materials in accordance with the tentative procedure recommended by the committee, and the other to investigate and secure, if possible, the necessary funds and personnel for establishing a fellowship at the National Bureau of Standards for the purpose of conducting further research in connection with the proposed tentative method.

Nomenclature and Definitions.—Considerable study has been given to the development of a definition for "incombustibility," and a draft of a proposed test has been under discussion. Testing laboratories were to conduct tests using this procedure and equipment in an effort to determine its usefulness. No conclusive results were attained due to the limited amount of testing that was done. The principal difficulty arises from trying to define "incombustibility." A different approach to the problem is now being considered and a definition for "combustibility" is under discussion. Several testing laboratories have agreed to conduct tests on various materials to provide data from which it is hoped a suitable definition can be decided upon. An advisory study group has been appointed to assist in the development of the necessary data.

This report has been submitted to letter ballot of the committee, which consists of 50 members; 46 members returned their ballots, of whom 43 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

A. L. BROWN.
Chairman.

H. M. ROBINSON,
Secretary.

REPORT OF COMMITTEE E-7
ON
NON-DESTRUCTIVE TESTING[1]

Committee E-7 on Non-Destructive Testing met at Atlantic City, N.J., on June 28, 1949, in conjunction with the Annual Meeting of the Society. Numerous Executive Council and subcommittee meetings have been held since.

The membership of the committee has increased during the year to 109, including 3 consulting members. The membership of Committee E-7 is not classified.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, J. H. Bly.
Vice-Chairman, H. E. Seemann.
Secretary, D. T. O'Connor.

TENTATIVE CONTINUED WITHOUT REVISION

In accordance with a special subcommittee's recommendation last year, it is recommended that the Tentative Industrial Radiographic Standards for Steel Castings (E 71 – 47 T) be continued in their present form for one more year.

GENERAL ACTIVITIES OF THE COMMITTEE

The papers delivered at the Committee E-7 technical meetings on Radiography and Ultrasonic Testing held during the Annual Meeting in 1949 have been collected into two publications which are now in process of final editing and printing. They should be available soon after the Annual Meeting in 1950.[2] Other general activities include: (*1*) Sponsorship during the Annual Meeting of a Symposium on the Rôle of Nondestructive Testing in the Economics of Production, and (*2*) Sponsorship of a Radiographic Section of the 1950 Photographic Exhibit, the object of which is to show correlations between different non-destructive tests or methods.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Recommended Radiographic Procedure (H. E. Seemann, chairman) has almost completed the initial work on its Proposed Tentative Recommended Practice for Radiographic Testing and hopes to present the preliminary document for Committee E-7 comment and trial at the Annual Meeting.

Subcommittee II on Radiographic Standards for Steel Welds (Alexander Gobus, chairman), charged with the responsibility of preparing specimens containing various discontinuities generally encountered in steel welds and making standard radiographs of the specimens, has reached agreement on the types and sizes of specimens to be prepared initially and on the radiographic methods to be employed. The sample preparation has been assigned to various volunteers who will also prepare radiographs. Further radiography, as necessary, will be assigned to other volunteers, and the specimens and final radiographs will be the property of the

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] Issued as separate publications, *STP No. 96*, July, 1950, and *STP No. 101*, January, 1951.

Society. Two of the volunteers report good progress in the sample-preparation phase; radiographs of one set of samples will be available for examination by the subcommittee at the time of the Annual Meeting.

Subcommittee III on Magnetic Particle and Fluorescent Penetrant Testing of Steel Castings (Hamilton Migel, chairman) has decided to limit its activities initially to magnetic particle testing of "large" steel castings (over 20 in. in any dimension), and to begin by preparing a set of specific recommended procedures giving detailed information on the procedure for inspection of such castings. This work is now under way, and the subcommittee hopes to have the preliminary document in fair shape by the time of the Annual Meeting.

Subcommittee IV on Collaboration with Committee E-11 (R. C. McMaster, chairman) had as its objective originally the sponsorship of a joint symposium in 1950 with Committee E-11. This turned out to be impracticable, since Committee E-11 is occupied this year with other considerations, and the Executive Council agreed on a symposium on the Rôle of Non-Destructive Testing in the Economics of Production, to be sponsored solely by Committee E-7, instead. H. H. Lester was appointed general chairman of this symposium, and the manifold problems of publicity, coordination, and specific arrangements were assigned to Subcommittee IV. This symposium, planned as a part of the 1950 Annual Meeting, has therefore been the project of the subcommittee for this year.[3]

This report has been submitted to letter ballot of the committee, which consists of 106 voting members; 76 members returned their ballots, of whom 70 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. BLY,
Chairman.

D. T. O'CONNOR,
Secretary.

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[3] In addition to the papers presented at this symposium, several other papers dealing with non-destructive testing were presented at the 1950 Annual Meeting. These were as follows:

C. H. Hastings, "An Evaluation of Radiography, with Particular Emphasis on Detection Methods." ASTM BULLETIN, No. 172, February, 1951, p. 66.

R. D. Kodis, "The Development of a Flaw Detector for Tubes," see p. 1196.

D. Berdan and R. K. Bernhard, "Pilot Studies of Soil Density Measurements by Means of X-Rays," see p. 1328

A. G. H. Dietz, G. M. Kavanagh, P. J. Closmann, and J. N. Rossen, "The Measurement of Dynamic Modulus in Adhesive Joints at Ultrasonic Frequencies," see p. 1414.

J. R. Leslie, "Pulse Techniques Applied to Dynamic Testing," see p. 1314.

REPORT OF COMMITTEE E-9

ON

FATIGUE[1]

In December, 1949, the Manual on Fatigue Testing was published.[2] It is believed that this book will be helpful to those setting up facilities for fatigue testing and to those responsible for the reporting of such tests. The section on nomenclature has been of wide interest, judging from correspondence. The excellent Headquarters' work of Carter S. Cole, who passed away November 17, 1949, was an important contribution to the Manual.

The Papers Review Subcommittee has been active throughout the year. A Session on Fatigue was arranged for the Pacific Coast Meeting in October, 1949, comprised of the following papers:

"Discussions of a Century Ago Concerning the Nature of Fatigue and Review of Some of the Subsequent Researches Concerning the Mechanism of Fatigue," R. E. Peterson, Westinghouse Electric Corp., East Pittsburgh, Pa.,[3]

"Fatigue Characteristics of Aircraft Materials and Fastenings," Thomas Piper, K. F. Finlay and A. P. Binsacca, Northrop Aircraft, Inc., Hawthorne, Calif.,[4]

"Fatigue Strength of Steel Through the Range from $\frac{1}{2}$ to 30,000 Cycles of Stress," M. H. Weisman and M. H. Kaplan, North American Aviation, Inc., Inglewood, Calif.,[5]

"Fatigue Notch Sensitivities of Some Aircraft Materials," H. J. Grover, Battelle Memorial Institute, Columbus, Ohio.[6]

In addition to the papers on Fatigue presented at the Pacific Coast Meeting, a Fatigue Session was held at the Annual Meeting. Committee E-9 was instrumental in providing a number of papers for this session.

H. F. Moore, chairman of Task Group on Effect of Speed of Testing on Fatigue Test Results of Committee E-1, gave a summary of this work at the meeting of Committee E-9. The report of the Task Group is printed as Appendix I to the 1950 Report of Committee E-1 on Methods of Testing.

A Survey Subcommittee was appointed, consisting of T. J. Dolan (chairman), J. A. Bennett, E. J. Greene, L. R. Jackson, and W. C. Lewis. The primary objectives of the subcommittee were:

1. To prepare a list of all fatigue projects that are now in progress in the principal laboratories of the United States and Canada.
2. To compile a list of fatigue topics on which further information is desired.

It was felt that this information would prove valuable in eliminating duplication of effort, and in calling attention to desirable types of research work that might serve as thesis projects in university research laboratories. Publication of the list of active projects should further serve to acquaint various agencies with laboratories currently conducting research of direct interest.

The report of the Survey Subcommittee is appended hereto. It consists essentially of two lists corresponding to the aforementioned objectives. The subcommittee is to be commended for carrying out an extensive project in an unusually short time. Some means, as yet undecided, will be devised for keeping the lists up to date.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] Issued as a separate publication, *STP No. 91*, December, 1949.

[3] ASTM BULLETIN, No. 164, February, 1950, p. 50 (TP62).

[4] ASTM BULLETIN, No. 166, May, 1950, p. 60 (TP122).

[5] See p. 649.

[6] See p. 717.

Committee E-9 has lost an outstanding member in the death of H. W. Gillett. His keen perception and clear, forceful writing have been of great value over the years. New committee members are: B. J. Lazan, J. Marin, J. C. Millson, D. A. Paul and R. C. A. Thurston.

This report has been submitted to letter ballot of the main committee which consists of 37 members; 29 members returned their ballots, of whom 28 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

REPORT OF SURVEY OF FATIGUE PROJECTS

In January, 1950, a general questionnaire was sent out to a selected list compiled by the subcommittee as follows:

A.—Government, association, or endowed research laboratories	44
B.—University laboratories	46
C.—Industrial and commercial research laboratories	131
Total	221

A total of 98 replies (44 per cent) have been returned thus far, of which 34 organizations indicate no current research in progress and 64 laboratories have furnished information regarding at least one current project.

In compiling the list of topics, appended hereto, on which information is needed, it was found that many organizations had made similar suggestions with only slight difference in emphasis; in other instances, some of the suggestions received were rather broad or vague. These have, therefore, been edited to combine similar projects or to clarify the suggestions somewhat. These lists are undoubtedly incomplete; some replies to the questionnaire are still being received. However, if it is deemed advisable, the lists could be revised or supplemented periodically and more complete coverage obtained.

Respectfully submitted on behalf of the survey subcommittee,

T. J. Dolan,
Chairman.

W. C. Lewis,
Secretary.

APPENDIX

CURRENT PROJECTS ON FATIGUE PROPERTIES OF MATERIALS

1. *Aberdeen Proving Ground, Ordnance Department, U. S. Army:*

(*a*) The effect of acid pickling and various inhibitors on the fatigue strength of low, medium, and high-carbon steels.

2. *Materials Laboratory, Air Materiel Command, A.A.F.:*

(*a*) Fatigue properties in bending and axial loading of unnotched and notched extruded ZK60 and AZ80 magnesium alloys.

(*b*) Statistical study of fatigue properties in reversed bending of 75S-T6 aluminum alloy rolled plate.

(*c*) Fatigue properties in axial loading for various mean stresses in S.A.E. 4340 and S.A.E. 8630 steels at room and elevated temperatures.

(*d*) Fatigue properties of inert-shielded tungsten-arc welded 14S-T and 75S-T6 aluminum alloy plates compared to unwelded material.

(*e*) Fatigue tests of turbine buckets at elevated temperatures.

3. *Propeller Laboratory, Air Materiel Command:*

(*a*) The fatigue strength of stainless steel rotor blade section of types suitable for helicopter use.

(*b*) The fatigue strength of blade shanks for X76S-T aluminum alloy propellers.

4. *Aluminum Company of America:*

(*a*) Rotating-beam fatigue properties of commercial aluminum and magnesium alloys as currently produced, particularly with reference to new alloys and tempers and to improvements in production methods.

(*b*) Fatigue strengths of commercial aluminum and magnesium alloys under various ranges of axial stresses.

(*c*) Fatigue strengths of commercial aluminum and magnesium alloys at elevated temperatures.

(*d*) Repeated-flexure fatigue strength of aluminum alloy sheet.

(*e*) Fatigue strengths of commercial aluminum and magnesium alloys in presence of sharp notches.

(*f*) Fatigue properties of commercial aluminum and magnesium alloys subjected to torsional stress.

(*g*) Effects of anodic coatings on the fatigue strength of aluminum alloys.

(*h*) Static and fatigue strengths of several high-strength aluminum alloy bolted joints.

(*i*) Fatigue strength of small joints in aircraft sheet.

5. *American Steel Foundries:*

(*a*) Fatigue strength of large hot-coiled helical compression springs of S.A.E. 1095 and 8600 steels.

(*b*) Effect of shot peening on fatigue strength of hot-coiled helical compression springs and of elliptic plates of S.A.E. 1095 steel.

(*c*) Effect of welding on fatigue strength of S.A.E. 1030 and 1330 steels.

(*d*) Effect of prestressing screw threads in 8640 and modified 1030-N steels.

6. *American Steel and Wire Co.:*

(*a*) The fatigue strength of various grades of galvanized steel wire subjected to reversed bending.

(*b*) Evaluation of various grades of music spring wire in the rotating-arc fatigue testing machine.

(*c*) Vibration fatigue characteristics of steel-reinforced aluminum cable and other composite types of electrical conductors under various tensile loads.

(*d*) Vibratory fatigue characteristics of various galvanized carbon steel and stainless steel 7-wire strands under various tension loadings and amplitudes.

(*e*) Fatigue characteristics of high and medium carbon strip steels in reversed bending.

7. *Armour Research Foundation:*

(*a*) Fatigue tests under progressive load, to determine endurance limit by means of a shorter and less expensive test.

8. *Baldwin Locomotive Co.:*

(*a*) The development of a high-temperature fatigue machine for simulated service testing of turbine bucket materials.

9. *Barnes-Gibson Raymond Division, Associated Spring Corp.:*

(*a*) The effect of temperature on the fatigue properties of steel spring wire.

10. *Battelle Memorial Institute:*

(*a*) Investigation of induction heating in relation to industrial gas heating—effects of case depth and metallurgical structure on the fatigue strength of S.A.E. 1045 steel.

(*b*) The effect of severity of surface grinding on the fatigue strength of high-hardness vanadium ball-bearing steel.

(*c*) The effect of notches on the direct stress fatigue strength of 24S-T, 75S-T, and S.A.E. 4130 aircraft sheet materials.

11. *Bell Telephone Laboratories, Inc.:*

(*a*) The influence of metallurgical variables on the flexural fatigue properties of phosphor bronze sheet.

(*b*) The flexural fatigue characteristics of lead and lead alloys.

12. *Beryllium Corp.:*

(*a*) The effect of heat treatment on the fatigue strength of beryllium copper wire.

13. *University of California at Berkeley:*

(*a*) The effect of stress frequency and temperature on the axial tension-compression fatigue strength of Inconel.

14. *University of California at Los Angeles:*

(*a*) A study of the effect of residual stress upon the bending fatigue strength of notched 61S-T aluminum plate, using X-ray diffraction stress measuring equipment.

(*b*) The effect of heat treatment on the bending fatigue strength of 0.020-in. beryllium copper sheet stock.

15. *Canadian Bureau of Mines:*

(*a*) The effect of various welding techniques on the fatigue strength of 5 per cent Cr, 0.5 per cent Mo steel pipe.

(*b*) The effect of clamping conditions on the fatigue strength of plain carbon steel leaf springs.

(*c*) The fatigue strength and dynamic creep properties of selected alloys at high temperature.

(*d*) The effect of shot peening, stress relieving, and super-finishing on the rotating-beam fatigue strength of selected steels.

(*e*) Results of rotating-beam fatigue tests to determine the optimum treatment for mining drill rod of S.A.E. 1080, Cr-Mo, and Ni-Cr-Mo steels.

(*f*) A study of factors affecting the fatigue notch sensitivity of various steels at different hardness levels.

16. *Case Institute of Technology:*

(*a*) The rotating-beam fatigue limit of cold drawn steel as a function of cold work.

17. *Civil Engineering Department, Columbia University:*

(*a*) The effect of intermittent rest periods at elevated temperatures on the fatigue life of butt-welded mild steel specimens in bending.

18. *Consolidated Vultee Aircraft Corp., San Diego Division:*

(*a*) Effect of repetitions of load at varying stress levels on the fatigue life and static strength of 24S-T sheet.

(*b*) Fatigue tests of riveted and bolted joints of 75S-T sheet.

19. *Cornell Aeronautical Laboratory, Inc.:*

(*a*) The effect of temperature on the relative fatigue life of some high-temperature alloys as determined with a modified G. E. Pneumatic Resonance Testing machine.

(*b*) The effect of stress concentrations on the fatigue life of aluminum aircraft alloys under axial loading, for high mean stresses.

20. *Douglas Aircraft Co., Inc.:*

(*a*) The fatigue strength of riveted or bolted joints or both in aluminum and magnesium alloys as affected by rivet or bolt size, pattern and sheet gage.

(*b*) Pre-stressing techniques to overcome the effect of fatigue concentrators.

21. *E. I. du Pont de Nemours and Co., Engineering Department:*

(*a*) A statistical study of the effect of anisotropy of forged S.A.E. 4340 steel on its fatigue strength under combined stresses.

22. *Ecole Polytechnique, Montreal:*

(*a*) The fatigue strength under tension-tension loading of single and multiple spot-welded sheets, with and without high pressure treatment.

23. *Flakice Corp:*

(*a*) The effects of temperature and corrosive media on the fatigue life of metal conveyor belts.

24. *Ford Motor Co.:*

(*a*) The measurement by X-ray diffraction of the depth of deformation layer in pressure-rolled bearings and fillets and its relation to fatigue.

25. *Frankford Arsenal, Ordnance Department, U. S. Army:*

(*a*) The effects of shock and vibrations of an aircraft on ordnance materiel.

26. *General Electric Co.:*

(*a*) Fatigue tests of completed parts and assemblies and the effect of surface treatments on their fatigue properties.

(*b*) Flexural fatigue strength of spot-welded joints under non-zero mean stress.

(*c*) A study of the effect of notches and shot-peening on the high temperature fatigue properties of S-816 and Timken alloy.

(*d*) A study of the fatigue properties of materials and high temperatures under non-zero mean stress.

(*e*) A study of the effect of rest periods and low-temperature anneals on the fatigue life of metals.

27. *The Glenn L. Martin Co.:*

(*a*) A study of possible methods of improvement of fatigue characteristics of 75S-T6 sheet by alteration of heat treatment procedure.

(*b*) The effect of residual stress from forming operations on fatigue strength of 75S-T6 extrusion.

28. *Goodyear Aircraft Corp.:*

(*a*) A fatigue study on riveted joints of balsa-aluminum sandwiches in shear and bending.

29. *Hamilton Standard Propellers:*

(*a*) A study of some variables which affect the fatigue strength of braze-steel composites.

30. *Harvard University:*

(*a*) The effect of cyclic stress history on the fatigue life of S.A.E. 1045 steel and 24S-T aluminum.

31. *Haynes Stellite Division, Union Carbide and Carbon Corp.:*

(*a*) Determination of high temperature fatigue properties of Haynes alloys Nos. 21, 25, 36, and 88.

32. *Hunter Spring Co.:*

(*a*) A study of factors influencing the fatigue life of "NEG'ATOR" springs.

(*b*) Fatigue tests on spring wire.

33. *International Harvester Co., Manufacturing Research:*

(*a*) Comparative fatigue properties of molybdenum- and chromium-bearing steels for ball bearing races and balls; effect of surface finish and corrosion.

34. *Department of Civil Engineering, University of Illinois:*

(*a*) The fatigue strength of bolted structural joints.

(*b*) A study of fatigue test results on metals subjected to cycles of repeated compressive loading.

(*c*) The effect of residual stresses on cumulative damage in fatigue.

35. *Mechanical Engineering Department, University of Illinois:*

(*a*) The effect of the heat treatment and metallographic structure upon the fatigue strength of carburized steels.

36. *Department of Theoretical and Applied Mechanics, University of Illinois:*

(*a*) The effect of superposition of stress-raisers on the rotating-beam fatigue strength of S.A.E. 4340 steel.

(*b*) The investigation of fatigue damage in cartridge brass by a recrystallization method.

(*c*) A study of the coaxing phenomena in fatigue of metals.

(*d*) A study of the relation between metallurgical grain size, degree of cold work, and the fatigue characteristics of cartridge brass.

(*e*) A study of the influence of cold work and residual stress on the torsional fatigue life of S.A.E. 2340 steel.

(*f*) A study of phenomena leading to fracture in regions in which the nominal repeated stresses are compressive.

(*g*) A study of slip lines and crystal breakup in fatigue of metals by means of the electron microscope.

(*h*) A critical study of notch-sensitivity in fatigue of metals.

(*i*) The effect of shape of cross-section on the flexural fatigue strength of metals.

(*j*) The effect of stress history on the fatigue behavior of 75S-T aluminum alloy.

(*k*) The effect of elevated temperature on the fatigue strength of polyester-fiberglass laminates.

(*l*) The effect of mean stress and notches on the fatigue strength of S.A.E. 4340 steel in bending and in torsion.

(*m*) The effect of state of stress and mean stress on the fatigue strength of metals.

(*n*) The effect of low temperature on the fatigue properties of polystyrene and 25S-T aluminum alloy.

o) A study of fatigue-testing methods used for plastics.

(*p*) The combined effect of mean stress and different states of stress on the fatigue strength of X76S-T aluminum alloy.

(*q*) Fatigue characteristics of plain concrete in flexure.

(*r*) The fatigue properties of steels used for railroad rails.

37. *Lehigh University:*

(*a*) The effect of prior plastic strain, heat-treatment, and welding on the fatigue strength of pressure vessel type steels.

38. *Lockheed Aircraft Corp.:*

(*a*) The fatigue strength of axially loaded high-strength alloy steels as affected by notch severity and specimen size.

(*b*) A study of the fatigue strength of aluminum alloys under distributed loading spectrums.

(*c*) The development of a fatigue failure warning indicator coupon to warn of approaching fatigue failure in aircraft structures.

39. *Menasco Manufacturing Co.:*

(*a*) The design, manufacture, and testing of a complete aircraft landing gear to withstand a definite number of cycles of repeated loading in the finite life range of fatigue.

40. *P. R. Mallory and Co., Inc.:*

(*a*) The fatigue strength of titanium alloys.

41. *North American Aviation, Inc.:*

(*a*) The fatigue properties of notched and unnotched specimens of titanium sheet subjected to tension-tension loading.

(*b*) Mechanical properties of stainless steel type 303 condition B.

42. *New York Naval Shipyard, U. S. Navy:*

(*a*) Fatigue study of most satisfactory methods of salvaging diesel engine crankshafts.

(*b*) The design and development of resonance-type fatigue test machines and automatic controls for tests in bending and torsion.

(*c*) A study of the influence of materials, methods of machining, and combined stresses on the fatigue life of crankshafts.

(*d*) Fatigue strength of large riveted or welded joints as used in structural members of naval vessels.

(*e*) Flexural fatigue strength and quality control tests of plastics, boat hull and canopy materials.

(*f*) Fatigue tests to evaluate adhesive strength of polymer-type caulking compounds to wood planking.

43. *Aeronautical Materials Lab., Naval Air Experimental Station, Navy Department:*

(*a*) Study of the effects of controlled overstress on the endurance strength of aluminum and magnesium alloys.

(*b*) The effect of flight stress on the mechanical properties of sheet taken from aircraft wing panels, particularly on fatigue strength.

(*c*) The comparative fatigue strength of various heat-resistant alloys in sheet form at elevated temperatures.

(*d*) Simulated service fatigue tests of turbine blade specimens at elevated temperatures.

(*e*) Comparative fatigue tests of cemented, riveted, and cemented-riveted metal joints.

(*f*) Flexural fatigue strength of high-strength glass fabric-polyester resin laminates for use in aircraft.

(*g*) Investigation to study shock absorber cords by repeated flexing for revision of Specification AN-C-175.

44. *U. S. Naval Engineering Experiment Station:*

(*a*) Fatigue as a method of evaluating various metallizing and electroplating procedures for reconditioning work on damaged machinery components.

(*b*) The fatigue properties of S.A.E. 4340 steel diesel engine crankshafts fabricated by welding.

(*c*) The corrosion fatigue properties of high-strength marine-propulsion shafting covered with an overlay of corrosion-resistant alloy.

(*d*) The effect of keyways on the torsional fatigue properties of steel propulsion shafting.

(*e*) The fatigue properties of high-strength propulsion shafting as affected by flame-hardening in localized areas.

(*f*) The fatigue properties of titanium and its alloys.

45. *Naval Research Laboratory:*

(*a*) The effect of fluctuating stresses on the finite fatigue life of metals at elevated temperatures under conditions of axial loading.

(*b*) The relation between internal friction or damping and the state of fatigue in metals.

(*c*) Rotating-beam fatigue properties of engineering materials in the finite life range of 1 to 20,000 cycles.

46. *Ohio State University:*

(*a*) Fatigue failure of metals subjected to biaxial stresses.

(*b*) A study of the effect of stress concentration on the fatigue life of metals.

(*c*) An experimental study of cumulative damage in fatigue of metals.

47. *Oklahoma Power and Propulsion Lab., Oklahoma A & M College:*

(*a*) Investigation of fatigue strength of alloy steel pin in shear.

(*b*) Investigation of static and fatigue strength of several commercially available adhesives for metal-to-metal bonding.

48. *Ontario Research Foundation:*

(*a*) A study of the effects of corrosive media and various conditions of dynamic loading on the fatigue life of wire rope.

49. *Pennsylvania Railroad:*

(*a*) Corrosion-fatigue of rail web steel in repeated bending.

50. *Pennsylvania State College:*

(*a*) The effect of elevated temperatures on the fatigue strength of 14S-T aluminum as determined with the Westinghouse high-temperature, vibration fatigue machine.

(*b*) The fatigue strength of Douglas fir in completely reversed bending.

(*c*) The effect of specimen size on the flexural fatigue strength of S.A.E. 1020 steel as compared with values predicted by Weibull's statistical method.

(*d*) The fatigue strength of Alcoa 14S-T aluminum alloy as affected by combined tension-tension fatigue stresses.

51. *Princeton Fatigue Testing Laboratory:*

(*a*) The effect of corrosion on the fatigue strength of steel and copper wires at room and elevated temperature.

(*b*) The fatigue strength of small steel cable subjected to pulsating tensile loads.

(*c*) Fatigue testing of automotive tires.

52. *Purdue University, Timken Roller Bearing Co., and Westinghouse Research Labs.; Size Effect Investigation:*

(*a*) The effect of specimen size on the shape and location of the *S-N* curve for S.A.E. 4140 steel under axial loading.

53. *Standard Steel Spring Co.:*

(*a*) An investigation of some factors affecting the fatigue life of leaf and coil steel springs.

54. *Syracuse University:*

(*a*) Damping capacity, elasticity and fatigue properties of temperature resistant materials at room and elevated temperatures.

(*b*) Creep and rupture characteristics of six temperature-resistant materials under static and dynamic forces at 1350 F. and 1500 F.

(*c*) Fatigue testing of commercial parts and materials for evaluating and rating serviceability.

(*d*) A study of the relations between damping capacity, dynamic modulus of elasticity and the fatigue properties of selected metals.

55. *Wayne University:*

(*a*) The effect of electrical energy input on the fatigue strength of aluminum under tension-tension loading.

56. *U. S. Forest Products Laboratory:*

(*a*) The effect of repeated bending loads on wood materials used for bridge stringers.

(*b*) The relative fatigue strengths of adhesive bonded metal-to-metal lap joints at room, elevated, and reduced temperatures.

(*c*) The relative fatigue strength of sandwich constructions for aircraft in shear.

57. *Metallurgy Division, National Bureau of Standards:*

(*a*) A study of the fundamental properties of metals which affect their notch sensitivity in fatigue.

(*b*) Detection of fatigue damage in 4130 steel by a study of surface stresses in statically loaded specimens by means of X-ray diffraction.

(*c*) The effect of chromium plating on the rotating beam fatigue strength of S.A.E. X4130 steel.

58. *Engineering Mechanics Section, National Bureau of Standards:*

(*a*) The fatigue characteristics of riveted joints fabricated from various high-strength aluminum alloys.

(*b*) The fatigue strength in completely reversed bending of several simplified wing beams fabricated from high-strength aluminum alloy sheet.

(*c*) The effects of a sequence of fatigue stresses having two maximum amplitudes on the fatigue life of high strength aluminum alloy sheet.

59. *Rock Island Arsenal:*

(*a*) The effect of surface finish on the fatigue life of high tensile, low-alloy rolled plate, in reversed bending.

60. *University of Washington:*

(*a*) Fatigue aspects of the stability and durability of bituminous paving mixtures as determined by repeated flexure at constant deflection.

61. *Westinghouse Research Laboratories:*

(*a*) The fatigue properties of wrought and cast gas turbine alloys at elevated temperatures.

(*b*) Range of stress under axial fatigue of 14S-T compressor rotor disk material.

(*c*) See item 52.

62. *Tube Turns, Inc.:*

(*a*) *S-N* Relationships for piping components and fittings (welding elbows, miter bends, forged fabricated tees, etc.) and flanges (all types).

63. *The Timken Roller Bearing Co.:*

(*a*) The fatigue strength of 9½-in. diameter, press-fitted, crank-pin assemblies as influenced by composition and heat treatment of several steels.

(*b*) The influence of press fitted wheels on the fatigue strength of full-size railroad passenger car axles made from quenched and tempered plain carbon steel.

(*c*) A study of the size effect on filleted shafts of normalized S.A.E. 1050 steel.

(*d*) A statistical study of the *S-N* curve for normalized and stress-relieved S.A.E. 1050 steel.

(*e*) The effect of heat-treatment on the fatigue strength of 7¼-in. diameter filleted shafts of S.A.E. 1050 steel.

(*f*) A study of the effect of residual stress on the fatigue strength of large steel shafts.

(*g*) Size effect study of plain specimens 5¼ and 0.30-in. diameter.

(*h*) The fatigue testing of full size production members such as crankshafts having 8-in. diameter journals, oil well tool joints, etc.

64. *Aluminum Laboratories Limited:*

(*a*) The rotating-beam fatigue strength of aluminum alloys as influenced by method and quality of surface finish.

(*b*) The effect of test-section radius on the fatigue strength of aluminum alloys.

65. *Department of Ceramic Engineering, University of Illinois:*

(*a*) Evaluation of the effect of refractory ceramic base coats on the fatigue strength and fatigue life of various metals and alloys which are used or which it is desired to use in aircraft power systems.

66. *Massachusetts Institute of Technology:*

(*a*) The fatigue of aircraft structures including the study of the effects of cyclic loading on the mechanical behavior of aluminum alloys.

67. *Carnegie Institute of Technology:*

(*a*) The anisotropy of rotating-beam fatigue properties of S.A.E. 4340 steel gun forgings.

(*b*) The statistical nature of the rotating-beam fatigue properties of S.A.E. 4340 steel forgings.

68. *Langley Aeronautical Laboratory, N.A.C.A.:*

(*a*) Investigation of size and shape effects on specimens representative of aircraft components and attempt to establish general relations that govern the phenomenon involved.

69. *National Advisory Committee for Aeronautics:*

(*a*) Effect of surface finish on fatigue at elevated temperatures.

70. *Central Engineering Division, Chrysler Corp.:*

(*a*) Determination of the fatigue properties of S.A.E. 4023 quenched and drawn or annealed to several hardness levels.

(*b*) Influence of "as cast" surfaces and of variations in heat treatment on the fatigue characteristics of two cast irons.

(*c*) Study of the fatigue properties of S.A.E. 4063 at several hardness levels.

(*d*) A study of the fatigue properties of isothermally treated steel and the influence of residual stresses on the test results.

(*e*) Effect of specimen size and number of stress cycles on the effective strength reduction factor in fatigue.

(*f*) Investigation of the statistical nature of fatigue data and determination of the minimum number of test points required to produce a reliable *S-N* curve.

71. *S.K.F. Industries, Inc.:*

(*a*) Study of endurance of simple test specimens of hardened steels to investigate possible correlation with endurance of ball bearings.

ITEMS IN FATIGUE FIELD ON WHICH INFORMATION IS NEEDED

Basic Fatigue:

(*a*) Corrosion:

(*1*) Effect of combustion gases.

(*2*) Mechanism of fretting corrosion or chafing fatigue.

(*b*) Cumulative damage:

(*1*) Effect of loading at levels below endurance limit on fatigue life at other levels of stress.

(*2*) Validity of cumulative damage theory.

(*3*) Basic information on cumulative damage.

(*4*) Effect of overstressing on subsequent fatigue behavior.

(*5*) Effect of cycles of varying stress as compared to cycles of uniform stress.

(*c*) Damping capacity:

(*1*) Correlation of damping characteristics with fatigue life and frequency of loading.

(*d*) Heat-treatment:

(*1*) Effect on fatigue strength of various materials.

(*2*) Effect on fatigue strength of plated parts.

(*3*) Fatigue properties of hardened steels having a hardness exceeding Rockwell C45.

(*e*) Resistance at elevated and reduced temperatures:

(*1*) Effect of testing frequency.

(*2*) Notch sensitivity at elevated and reduced temperatures.

(*3*) Correlation of notch sensitivity in fatigue with that of impact and for static conditions at elevated temperatures.

(*4*) Effect of range of stress.

(*5*) Creep in fatigue specimens at elevated temperatures.

(*6*) Fatigue of heat-resistant alloys at elevated temperatures.

(*7*) Effects of temperature gradients such as are experienced in gas turbines and blast-driven turbo pumps.

(*f*) Fatigue crack:

(*1*) Propagation.

(*2*) Initiation.

(*3*) Residual stresses at tip of crack.

(*4*) Method of detecting damage prior to formation of crack.

(*5*) Correlation between magnetic properties and progress of failure.

(*g*) Loading:

(*1*) Effect of wave form.

(*2*) S-N curves between 1 and 50,000 cycles (both notched and plain specimens) for aircraft materials.

(*3*) Effect of speeds of less than 50 cycles per minute on S-N curves.

(*4*) Relation between conventional data and simulated test data obtained by high *versus* low frequency loading superimposed with interrupted loads.

(*h*) Material:

(*1*) Fatigue properties for pure or commercially pure aluminum.

(*2*) Fatigue properties in axial loading for plain and notched extruded sections of aluminum and magnesium alloys.

(*3*) Fatigue behavior in the plastic range.

(*4*) Reverting of hard-drawn copper to soft condition under variable stress.

(*5*) Effect of stress relief.

(*i*) Notch sensitivity:

(*1*) Effect of shape of notch.

(*2*) Effect of severity of notch.

(*3*) Effect of local plastic flow.

(*4*) Effect of grain size in area around notch.

(*5*) Correlation of fatigue concentration factors with those obtained by other methods.

(*6*) Effect of notches induced by impact.

(*j*) Shape of specimen:

(*1*) Effect of shape of cross section on axial and flexural fatigue.

(*2*) Effect of size and geometry on chromium-plated parts.

(*3*) Effect of size on fatigue behavior.

(*k*) Stress level:

(*1*) Behavior of materials at different levels (load spectrum).

(*2*) Fatigue life for fluctuating loads at high stress levels.

(*3*) Influence of combined stresses on fatigue strength.

(*l*) Surface or near surface finish or treatment on fatigue:

(*1*) Structure.

(*2*) Hardness.

(*3*) Decarburization.

(*4*) Rolling (effective depth).

(*5*) Plating (basic causes for reduction in fatigue life).

(*6*) Surface coatings on fatigue life of aluminum alloys.

(*7*) Grinding.

(*8*) Shot or sand blasting on fatigue life of extrusions or machined aluminum alloys.

(*9*) The effect of residual surface stresses having a high stress gradient.

(*m*) Theoretical considerations:

(*1*) Structural action preceding fatigue damage.

(*2*) Nature of fatigue phenomena.

Fundamental Problems for Aircraft:

- (*a*) Magnitude and types of fatigue problems that exist in aircraft.
- (*b*) Frequency spectrum of the vibrations at various locations in an airplane in flight.

Simple Machine Parts:

(*a*) Cap screws:

(*1*) Basic fatigue data.

(*2*) Effect of cold working.

(*3*) Effect of heat-treatment.

(*4*) Combination of bending and tension forces on screw.

(*b*) Springs:

(*1*) Effect of residual stress on fatigue behavior.

(*2*) Spring materials in fatigue in bending or in torsion (at other than room temperatures).

(*3*) Effect of shot-peening while under stress.

(*4*) Fatigue of helical spring washers.

(*c*) Tubing:

(*1*) Effect of wall thickness on fatigue behavior.

(*2*) Comparison of fatigue results of tubing *versus* solid bars.

(*3*) Influence of fillets, flair, and external notches on fatigue strength.

(*4*) Heavy-wall tubular sections, short life tests (100,000 cycles of stress or less).

(*d*) Wire:

(*1*) Fatigue of grooved copper and bronze trolley wire, and correlation with service stresses.

Miscellaneous:

- (*1*) Rubber isolation mountings for instruments. Method of determining damage prior to failure.
- (*2*) Timber fasteners (nails, bolts, screws, connectors) under repeated loads.

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS[1]

During the past year Committee E-11 on Quality Control of Materials has completed work on the first three sections of the A.S.T.M. Manual on Quality Control of Materials. These sections constitute revisions of the present A.S.T.M. Manual on Presentation of Data with its Supplements A and B. In addition to this, task groups have been working on a survey of A.S.T.M. sampling plans, recommended practices for the planning of inter-laboratory tests, procedures for determining the number of tests for a desired precision of an average, the sampling of bulk materials, and the fitting of curves for a linear relationship. A new task group has been authorized to study problems connected with the use of terms such as "precision," "accuracy," "reproducibility," "reliability," and allied terms. Committee E-11 has given assistance to other A.S.T.M. committees and members on problems covering special phases of application of statistical methods to A.S.T.M. problems.

The election of officers for the ensuing term of two years resulted in the selection of the following:

Chairman, H. F. Dodge.

Vice-chairman, A. E. R. Westman.

Secretary, O. P. Beckwith.

C. A. Bicking and E. G. Olds were elected members of the Advisory Committee.

Recommendations Accepted by the Administrative Committee on Standards

Subsequent to the 1949 Annual Meeting, Committee E-11 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (E 29 - 48 T) be revised. This recommendation was accepted by the Standards Committee on September 22, 1949, and the revised tentative recommended practices appear in the 1949 Book of A.S.T.M. Standards, Parts 1, 2, 3, 4, 5, and 6.

Adoption of Tentative as Standard

Committee E-11 recommends that the Tentative Recommended Practices for Designating Significant Places in Specified Limiting Values (E 29 - 49 T) be approved for reference to letter ballot of the Society for adoption as standard with an editorial change substituting "conformance to" for "compliance with" throughout the text.

The recommendation appearing in this report has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.

Publication of Manual on Quality Control

The committee recommends for publication Sections 1, 2, and 3 of the A.S.

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[1] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

T.M. Manual on Quality Control of Materials to replace the present A.S.T.M. Manual on Presentation of Data and Supplements A and B.[2]

Section 1 of the A.S.T.M. Manual on Quality Control of Material represents a relatively minor revision of the main section of the present A.S.T.M. Manual on Presentation of Data. It discusses the application of statistical methods to the problems of:

(*a*) The condensation of the information contained in a single set of "*n*" observations, and

(*b*) The presentation of the essential information in a concise form.

Section 2 of the A.S.T.M. Manual on Quality Control of Materials represents a moderate revision of Supplement A of the present A.S.T.M. Manual on Presentation of Data. It discusses the problem of presenting limits to indicate the uncertainty of the average, $\bar{X}$, of a unique sample of "*n*" observations and suggests a form of presentation for use when needed in A.S.T.M. reports and publications.

Section 3 of the A.S.T.M. Manual on Quality Control of Materials is an extensive revision of Supplement B of the A.S.T.M. Manual on Presentation of Data. The technical content of the replaced publication is retained, but additions and modifications are introduced. The principal changes are:

(*a*) Reversal of the order of presentation in the earlier publication, giving first "Control—No Standard Given" and second "Control with Respect to a Given Standard."

(*b*) Separate treatment of control charts for "Number of Defectives," "Number of Defects," and "Number of Defects per Unit."

(*c*) Addition of material on control charts for individuals, as requested by members of the Society.

(*d*) Addition of appendices giving explanatory notes, a glossary of terms and symbols, tables and formulas found useful for reference purposes.

This report has been submitted to letter ballot of the committee, which consists of 22 members; 20 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. DODGE,
Chairman.

O. P. BECKWITH,
Secretary.

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[2] Issued as a special technical publication, January, 1951, (*STP 15-C*).

TECHNICAL PAPERS

With Discussions

EDGAR MARBURG LECTURE

THE PURPOSE of the Edgar Marburg Lecture is to have described at the Annual Meetings of the American Society for Testing Materials, by leaders in their respective fields, outstanding developments in the promotion of knowledge of engineering materials. Established as a means of emphasizing the importance of promoting knowledge of materials, the Lecture honors and perpetuates the memory of Edgar Marburg, first Secretary of the Society.

CHEMICAL SPECTROSCOPY[1]

TWENTY-FOURTH EDGAR MARBURG LECTURE

BY WALLACE R. BRODE[2]

In previous Marburg Lectures, such as G. L. Clark's (1)[3] discussion on X-rays and C. E. K. Mees' (2) discussion on color, there have been presented some of the basic concepts of radiant energy and its measurement. In this presentation I shall endeavor to extract from these and other earlier discussions (3, 4) some of the basic concepts presented and to weave them into a survey on the application of spectroscopy to chemistry.

Spectroscopy is the science of separation of radiant energy, within prescribed limitations, into an ordered arrangement according to wavelength or frequency, and a study of the resulting monochromatic or limited frequency radiation. *Chemical spectroscopy* is, more precisely, the use of spectroscopy in the solution of chemical problems, such as qualitative and quantitative analysis, and a determination of the physical state or nature of combination in molecules and their mixtures.

Generally, some physical property is used to determine the identity or character of a material, and in a systematic analysis procedure the materials are arranged in order of ascending or descending changes in this physical property. These properties may be purely mechanical, such as weight and viscosity; electrical, such as conductivity and capacity; thermal, such as melting point and thermal conductivity; or optical, such as refractive index and rotatory dispersion. Physical properties may be additive with the physical effects of each property combining to produce an effect which is an average, such as refractive indices or rotation—where there is no interaction between the different molecules and one substance is merely the diluent of the other.

Other properties, however, are not additive but rather constitutive and remain as separate effects characteristic of the specific substance, even in mixtures—such effects would be X-ray and mass spectra diffractions. The fields of emission and absorption spectroscopy involve such constitutive properties, in which spectra of certain components retain their individuality in mixtures.

In this discussion an attempt is made to survey briefly the extensive field of the application of spectroscopy to chemical analysis. This is a major undertaking, as thousands of research articles and many books (5 to 10) have been written on this subject, especially in the past decade.

While the term spectroscopy has been variously used to represent the ordered arrangement of any group of substance or effects, such as a spectrum of people, elements, or materials, we would prefer to confine it to its radiant energy concept, which would eliminate even mass spectra data from our discussion; in fact this discussion will be further restricted to those regions of radiant energy which lie between the far infrared and the far ultraviolet.

Spectroscopy has proven to be one of the most powerful of our modern research tools in probing our mechanical universe from the submicrostructure of atoms and molecules to the macro phenomena of temperature, speed, and direction of motion of our stellar bodies. The principal users of spectroscopy have been the chemists and physicists, in fact, the earliest applied developments in spectroscopy were by chemists and are considered to date from the time of Kirchhoff and Bunsen (11) in 1860. Inability to apply spectroscopy to more precise determination led to its gradual abandonment by chemists in the latter part of the Nineteenth Century, and even Kayser in his "Handbuch der Spektroskopie" (12) in 1910, concluded that qualitative spectroscopic analysis was not too useful and that quantitative spectroscopic analysis was impractical.

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[1] Read on June 27, 1950, before the Fifty-third Annual Meeting of the Society, Atlantic City, N. J.

[2] Associate Director, National Bureau of Standards, Washington, D. C.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 557.

As a good example of the interdependence of the advance of one field of science upon the advances of another, the work of physicists in what might be called physical spectroscopy, that is, theory of spectra and spectral energy analysis and photography, was essential to provide the chemists and applied physicists with the required information which was lacking in the 1870–1880's and which, when finally available in the 1910–1920's, provided the basis for the application of spectroscopy to chemical analysis. Our modern quantitative spectroscopic analysis and the expansion of this science dates from the pioneering work of de Gramont (13) in this period and perhaps more precisely from the publication from the National Bureau of Standards by Meggers, Kiess, and Stimson (14) on "Practical Spectrographic Analysis" in 1922.

The field of chemical spectroscopy is of particular interest to the American Society for Testing Materials where emphasis is placed on quality specification and improved methods of analysis and testing. As will be mentioned subsequently in this presentation, A.S.T.M. has played no small part in the last quarter century in the development of this field of chemical spectroscopy. Equally proud of its contribution to the field of chemical spectroscopy is the institution which I represent, the National Bureau of Standards, which has cooperatively supported the actitivies of the A.S.T.M. in this and other fields.

In confining this discussion to chemical spectroscopy it is necessary to omit, in the interest of brevity, the earlier historical and development work in both chemical and physical spectroscopy and to proceed at once to the technical consideration of the subject. A somewhat elementary approach has been employed, which it is hoped will be indulged so that the subject will be more easily followed as a fairly rapid coverage of it is presented. In what is more or less a standard procedure of subdivision two separate fields of chemical spectroscopy will be considered—(*a*) emission and (*b*) absorption. In the emission field qualitative and quantitative elemental analysis will be considered, and in absorption, the infrared, visual, and ultraviolet absorption spectra as applied to the identification of atomic combinations and molecules.

EMISSION SPECTROSCOPY

General:

The theories of atomic structure which have been developed in the present century form the background for our explanation of the observed emission and absorption spectra (15 to 20). It will not be necessary, in a discussion of an applied science such as chemical spectroscopy, to acquire a full understanding of these theories so as to apply the principles involved. In a broad generalization it may be accepted as axiomatic that atoms are composed of positive cores made up of protons and neutrons and surrounded by shells or levels of electrons, the number and locations of which are fixed by certain laws of physics.

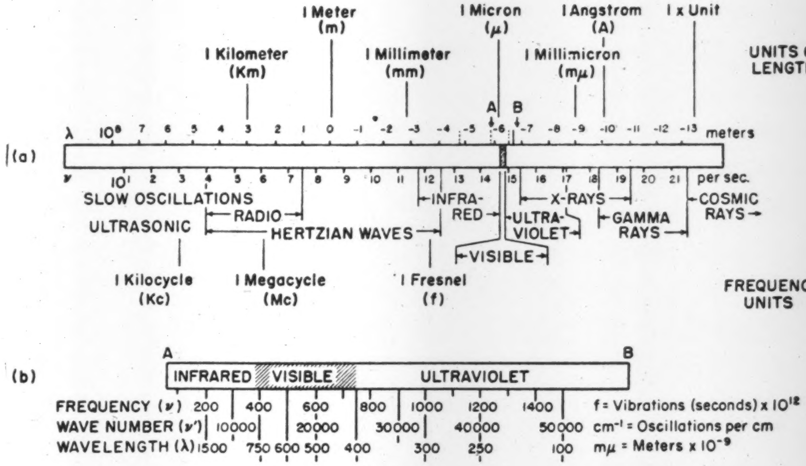
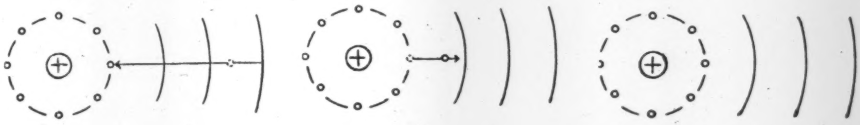


FIG. 1.—Graphic Indication of the Spectral Distribution of Radiant Energy.



(a) (b) (c)

FIG. 2.—Qualitative Representation of (*a*) Emission, (*b*) Absorption, and (*c*) Normal State.

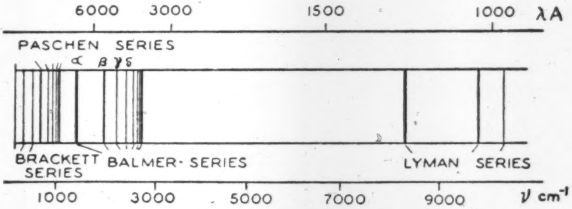


FIG. 3.—Hydrogen Spectrum.

By application of energy, which may be in the form of heat, light, or electricity, an electron may be displaced from what might be termed its stable level to some outer level of higher energy state. If sufficient energy is applied the electron may be removed from the atom, thus leaving an ion with a positive charge resulting from the loss of an electron. The excited atom produced by pushing out of an electron may revert to the normal state by loss of energy resulting from the return of the electron to its normal position; this return may take place in a single movement from the excited state or the electron may move to intermediate positions or shells in the process of return. Each movement results in the loss of energy and the energy is emitted in the form of radiant energy. Since each movement is in accordance with definite laws, these discrete energies may be recorded as of different frequencies since there is a definite relationship between the frequency or wavelength of radiant energy and the equivalent voltage or charge (Fig. 2).

Simple atoms like hydrogen (Fig. 3) will show a limited number of series of lines in their spectra, corresponding to progressive shifts of electrons in excited atoms as they revert to their normal position. The more complex the electronic structure of the atoms the greater the variety of lines produced in the emitted spectra, although there will appear to be a certain order or similarity in the spectra of certain electronically similar elements (21).

Elements with only one electron in the outer shell, as in the case of Na, K, etc., can be easily excited. The characteristic yellow sodium flame is produced by the heat of an ordinary flame, whereas in the case of those elements in which there is a completed electron shell, such as neon, a high voltage is required to displace an electron. The ease of ionization, or electrical potential that is required for removal of the first electron (Fig. 4), shows this periodic relationship and the spectra produced in the return of the electron to the normal atom is referred to as the neutral or I spectra. The second electron to be removed, in

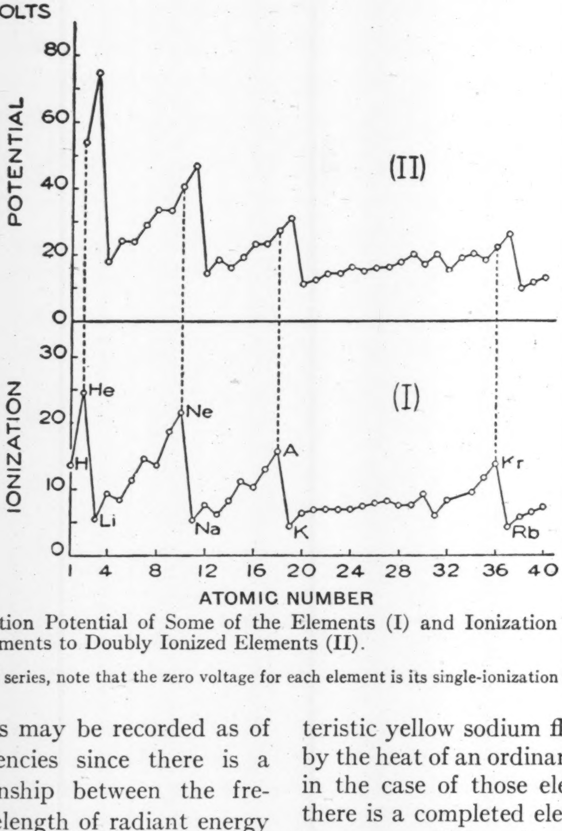
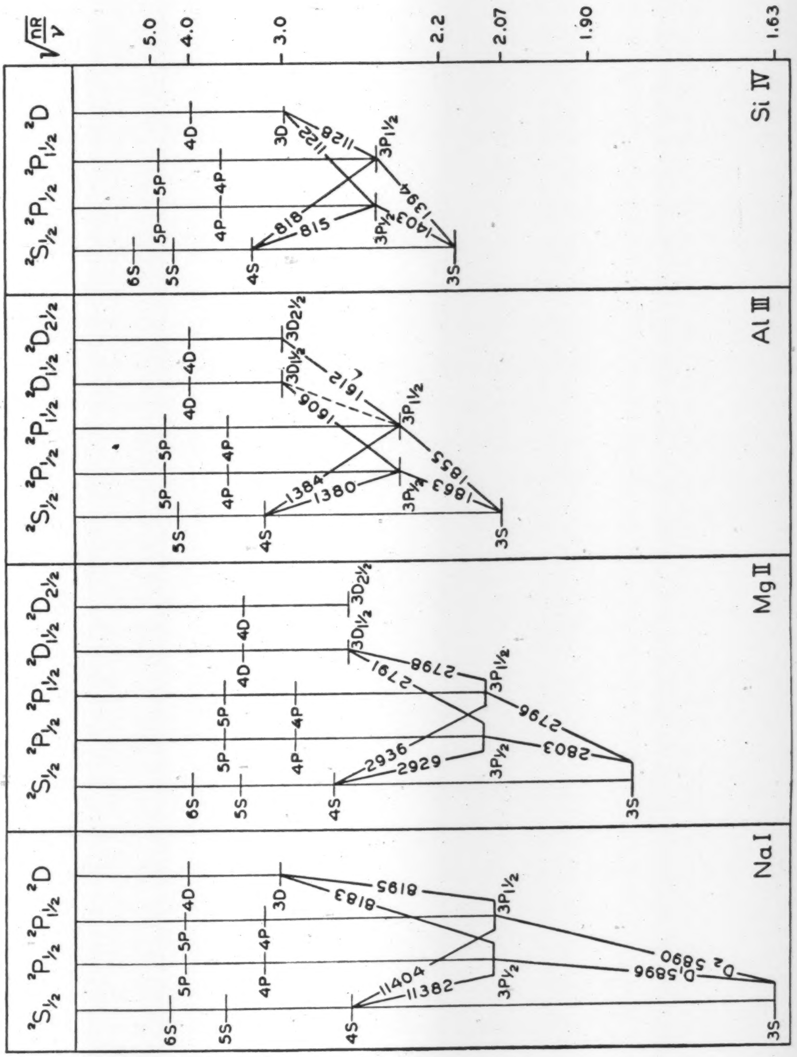


FIG. 4.—Ionization Potential of Some of the Elements (I) and Ionization Potentials of these Singly Ionized Elements to Doubly Ionized Elements (II).

In the II series, note that the zero voltage for each element is its single-ionization potential.


$^2S_{1/2}$
$^2P_{1/2}$
$^2P_{1\frac{1}{2}}$
^2D
$^2D_{1\frac{1}{2}}$
$^2D_{2\frac{1}{2}}$
$\sqrt{\frac{nR}{\nu}}$
5.0
4.0
3.0
2.2
2.07
1.90
1.63
Na I
Mg II
Al III
Si IV
6S
5S
4S
3S
5P
4P
4D
3D
$3P_{1/2}$
$3P_{1\frac{1}{2}}$
$3D_{1\frac{1}{2}}$
$3D_{2\frac{1}{2}}$
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11382
8195
8183
D_1 5896
D_2 5890
2936
2929
2798
2791
2803
2796
1384
1380
1606
1612
1863
1855
818
815
1128
1122
1403
1394

FIG. 5.—Level Diagrams of Four Spectra Arising in a System of Eleven Electrons, Each System Having a Different Nuclear Charge.

Ionization potential for NaI = 5.12, Mg II = 15, Al III = 28.3, and Si IV = 44.95 v.

returning to its normal position results in what is known as II spectra, and successive spectra provided through loss of additional electrons result in III, IV, and V spectra. In some of the high-temperature stars, spectra have been identified of the order of XV or higher.

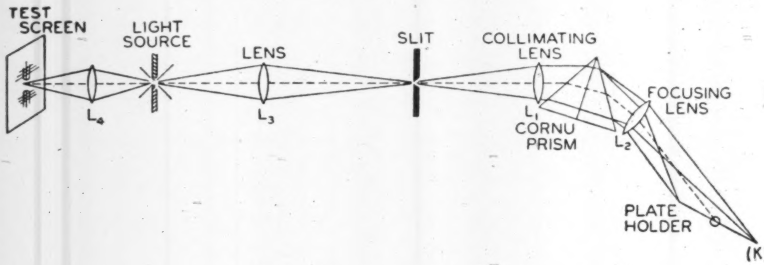

FIG. 6.—Optical Diagram of a Spectrograph.

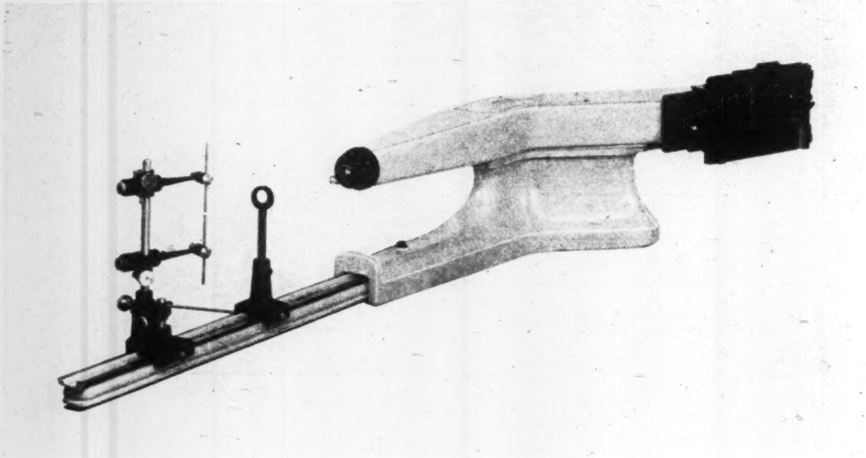

FIG. 7.—Hilger Medium Spectrograph.
60-cm. focal length, quartz Cornu prism

From Fig. 5 and a comparison of the indicated adjacent elements in the periodic table, it will be noted that the four elements, Na I, Mg II, Al III, and Si IV, (which indicate un-ionized sodium, magnesium with one electron removed, etc.) all have an identical shell of 23 electrons. They differ, however, in that they each have successively greater positively charged cores; hence it would be expected that each of their spectra would be similar but that those with a greater positive charge would require greater energy to push out the electron and conversely a greater energy would be released on the return of the electron. One can thus understand how, by relative comparison of spectra in stars with a shifted spectra of known elements, it might be possible to identify the spectra of energy levels that have not as yet been reached in our laboratory scale of operation.

Instruments:

Spectrographic equipment must disperse according to wavelength the light emitted by the sample, resulting in what is termed a spectrum or an ordered arrangement of radiant energy. The dispersion of radiant energy can be accomplished through the use of a grating

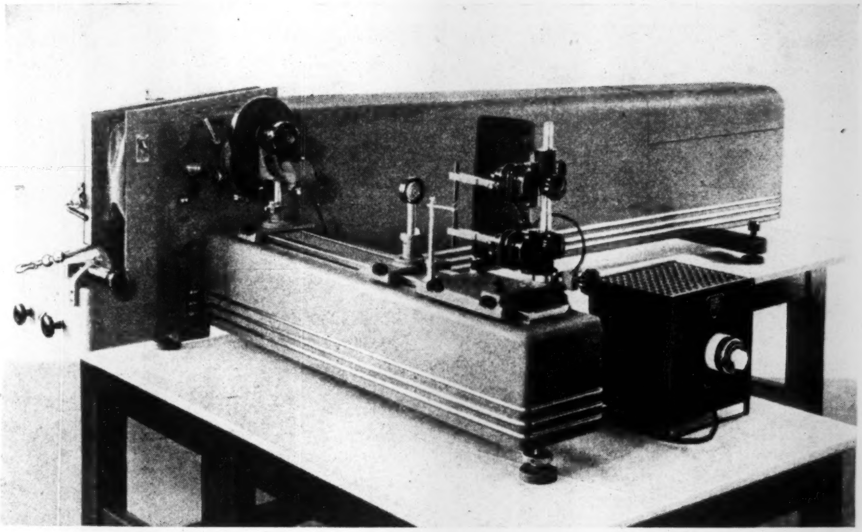

FIG. 8.—Bausch & Lomb Large Littrow Spectrograph and Illuminating Unit.
Focal length is 182.7 cm.

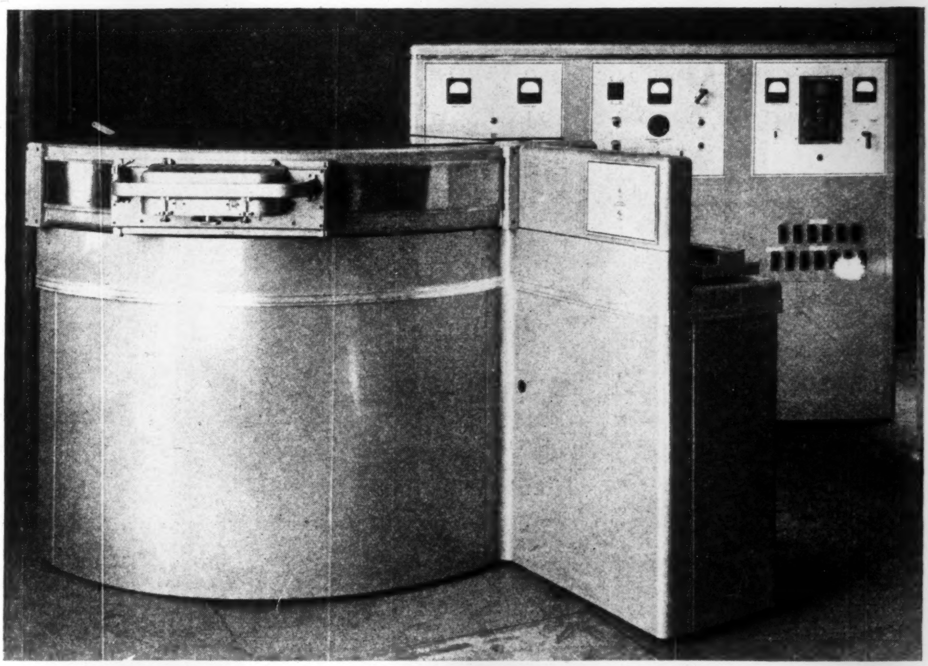

FIG. 9.—Applied Research Laboratories 2-m. Spectrograph—High Precision Source Unit.

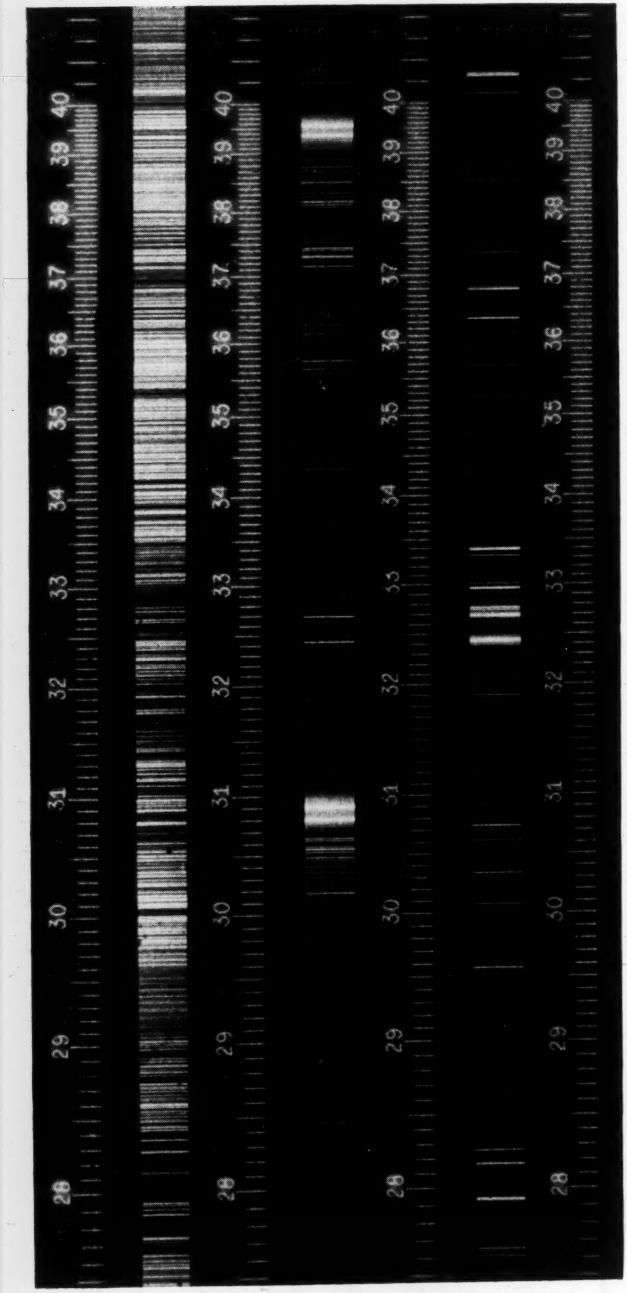
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FIG. 10.—Spectrograms of (top) Steel; (middle) Aluminum Alloy; (bottom) Brass.

which consists of fine lines on a reflecting medium, or a prism of refracting material such as glass or quartz. The basic spectrographic arrangement (Fig. 6) involves a source, focusing lens, slit, collimating lens, prism (or grating), focusing lens, and the receiver which may be a photographic plate, eyepiece or photocell. To economize in instrument size and refracting material such as crystalline quartz, a Littrow modification (Fig 8) of the prism spectrograph (Figs. 7) has been developed and has proven very satisfactory. In recent years there has been an extensive use of gratings (Fig. 9) in place of prism refraction material, although the synthetic production of crystalline material is offering the possibility of additional refraction materials. A typical photographic recording of a spectrograph, which is termed a spectrogram, is shown in Fig. 10.

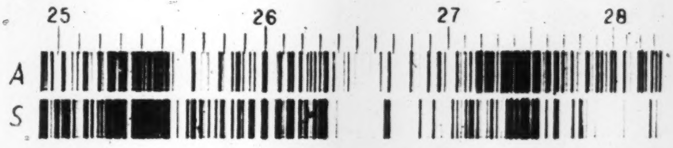


FIG. 11.—A Comparison of Arc (upper) and Spark (lower) Spectra of Iron. Shows Clearly the Importance of Source Control.

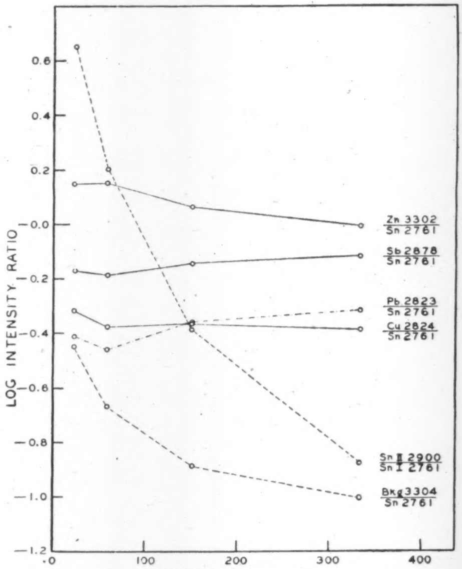


FIG. 12.—The Effect of Inductance on Intensity Ratios.

The spectrum and its notation are shown in Fig. 1 in a linear wavelength presentation although it can be seen that there is a similar reciprocal relation between wavelength and frequency. The portion of the spectrum (Fig. 1 (*b*)) which is primarily used for spectrochemical analysis is indicated as *A* to *B* and can be subdivided into infrared, visible, and ultraviolet. For our discussion we will consider only the visible and ultraviolet spectra with respect to emission analysis.

Qualitative Analysis:

Qualitative and quantitative spectrochemical analyses were considered in the late Nineteenth Century to be impractical because of the complexity of spectra and the variation in the spectra of elements depending on the nature of the light source used. However, with the development of an ordered explanation of the origin of spectra and the allocation of spectral lines into series (22), the identification of spectral lines from different elements has evolved. Certain lines are more intense than others, and some so intense that they remain visible even after considerable dilution of the element

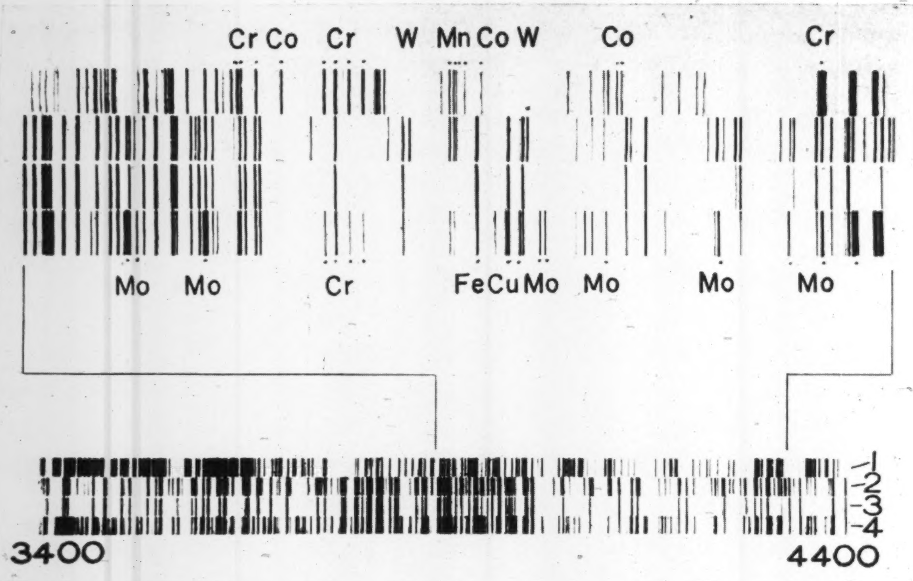
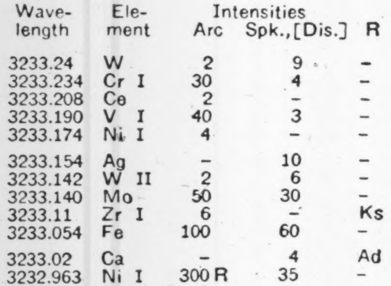


FIG. 13.—Qualitative Analysis for (1) Non-ferrous Alloy, (2) Co-W-Steel, (3) Iron, and (4) Molybdenum-Steel.



Wave-length	Ele-ment	Intensities Arc	Spk., [Dis.]	R
3233.24	W	2	9	–
3233.234	Cr I	30	4	–
3233.208	Ce	2	–	–
3233.190	V I	40	3	–
3233.174	Ni I	4	–	–
3233.154	Ag	–	10	–
3233.142	W II	2	6	–
3233.140	Mo	50	30	–
3233.11	Zr I	6	–	Ks
3233.054	Fe	100	60	–
3233.02	Ca	–	4	Ad
3232.963	Ni I	300 R	35	–
3232 875	Ce	3	–	–
3232.874	Co I	60	25	–
3232.80	Kr I	–	[2]	Me
3232.793	Cb	–	2 h	–
3232.791	Ti I	8	–	–
3232.791	Fe II	–	50	–
3232.78	I	–	[5]	Bl
3232.751	Ru I	50	4	–

FIG. 14.—Portion of a Page from the M.I.T.—Harrison 100,000 Line Tables (24) Arranged According to Wavelength.

by other elements. Such lines are called *raies ultimes* or persistent lines, and the accepted procedure in qualitative analysis is to examine the unknown spectra for the persistent lines of the known elements rather than to attempt to identify unknown weak lines in the spectra, which may be uncharted weak lines of the principal constituent rather than strong lines of the elements present in small amount.

Tables are available which list the observed lines of the source elements. Some of these tables are arranged by element, and others are arranged by wavelength (23, 24) (Fig. 14). These tables and the more condensed tables of the principal or persistent lines (Tables I and II) provide the essential data to detect the presence of various elements (5, 25 to 29). Most handbooks and applied spectroscopy books contain tables of persistent lines which are usually derived from the tables of de Gramont, although some have been developed to supply most persistent lines over a limited spectral range. On account of the varied and large number of lines produced by many elements, photographic charts of different element

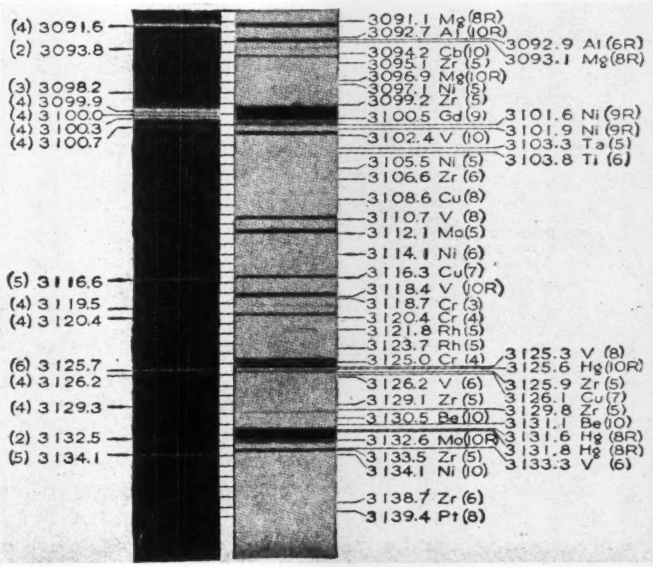
TABLE I.—PORTION OF A TABLE OF PERSISTENT LINES ARRANGED ACCORDING TO WAVELENGTH (5).[a]

Wavelength, Å	Element	Wavelength, Å	Element	Wavelength, Å	Element	Wavelength, Å	Element
3134.72	Hf II	3405.12	Co I	3642.78	Sc II	4012.39	Ce
58.87	Ca II	06.66	Ta	46.20	Gd	19.18	Th
63.40	Cb II	′14.77	Ni I	50.15	Hg I	20.40	Sc I
′75.04	Sn I	21.24	Pd I	53.50	Ti I	23.69	Sc I
79.33	Ca II	′34.89	Rh	54.83	Hg I	′30.76	Mn I
83.41	V II	36.74	Ru I	57.99	Rh I	32.98	Ga I
83.98	V II	37.02	Ir I	63.28	Hg I	33.07	Mn I
85.40	V II	38.23	Zr II	72.58	U	34.49	Mn I
94.98	Cb II	51.41	B II	83.47	Pb I	4040.76	Ce II
3215.56	W I	′53.51	Co I	92.36	Rh I	44.14	K I
′20.78	Ir I	60.47	Re I	92.65	Er	45.98	Dy

[a] The most persistent lines are indicated by a prime (′) mark

TABLE II.—EXAMPLE OF TABLE OF PRINCIPAL LINES BY ELEMENTS (5).
ALUMINUM, Al

Wavelength	Arc	Spark	Wavelength	Arc	Spark	Wavelength	Arc	Spark
II 2 094.8	. . .	50	I 2 378.4	40	20	I 3 082.2p	800	800
I 2 168.8	8R	12	2 568.0	200R	80R	I 3 092.7p	1000	1000
I 2 174.0	8R	10	2 575.1	200R	80R	3 092.8	50R	18
2 204.6	10R	10	2 575.4	30	30	II 3 586.9	. . .	500
I 2 210.0	12R	15	I 2 652.5	150R	60	I 3 944.0p	2000	1000
I 2 259.9	8	. . .	I 2 660.4	150R	60	I 3 961.5p	3000	2000
I 2 263.5	60R	25	II 2 669.2	3	100	II 4 962.1	15	3
I 2 269.1	60R	25	II 2 816.2	10	100	I 5 557.0	15	. . .
I 2 269.2	15R	. . .	3 050.1	18	10	I 5 557.9	15	. . .
I 2 367.1	150R	50R	3 054.7	20	10	II 6 231.8	. . .	30
I 2 **372.1**	18	10	3 057.2	15	18	II 6 243.4	. . .	100
I 2 **373.1**	100R	30	3 064.3	20	20	II 6 696.4	10	. . .
I **2** 373.4	200R	100R	3 066.2	25	25	6 698.8	10	. . .
						7 836.9	15	



(4) 3091.6
(2) 3093.8
(3) 3098.2
(4) 3099.9
(4) 3100.0
(4) 3100.3
(4) 3100.7
(5) 3116.6
(4) 3119.5
(4) 3120.4
(6) 3125.7
(4) 3126.2
(4) 3129.3
(2) 3132.5
(5) 3134.1
3091.1 Mg(8R)
3092.7 Al (10R)
3092.9 Al (6R)
3093.1 Mg(8R)
3094.2 Cb(10)
3095.1 Zr (5)
3096.9 Mg(10R)
3097.1 Ni (5)
3099.2 Zr (5)
3100.5 Gd (9)
3101.6 Ni (9R)
3101.9 Ni (9R)
3102.4 V (10)
3103.3 Ta (5)
3103.8 Ti (6)
3105.5 Ni (5)
3106.6 Zr (6)
3108.6 Cu(8)
3110.7 V (8)
3112.1 Mo(5)
3114.1 Ni (6)
3116.3 Cu(7)
3118.4 V (10R)
3118.7 Cr (3)
3120.4 Cr (4)
3121.8 Rh(5)
3123.7 Rh(5)
3125.0 Cr (4)
3125.3 V (8)
3125.6 Hg(10R)
3126.2 V (6)
3125.9 Zr (5)
3126.1 Cu(7)
3129.1 Zr (5)
3129.8 Zr (5)
3130.5 Be(10)
3131.1 Be(10)
3131.6 Hg (8R)
3132.6 Mo(10R)
3131.8 Hg (8R)
3133.5 Zr (5)
3133.3 V (6)
3134.1 Ni (10)
3138.7 Zr (6)
3139.4 Pt (8)

FIG. 15.—Use of Cards for Identification of Elements.

The unknown spectrogram is projected with enlargement to the same scale as the card spectrum. In this example, the unknown is an iron-containing compound and hence no adjacent iron spectrum is required.

spectra are available and are useful as comparisons in the identification of unknowns (5, 27, 29, 30) (Fig. 15). Some comparison charts have indicated positions of principal lines (elements to facilitate rapid qualitative analysis) (31). The "scale" usually used on these charts is an iron spectrogram which, while not providing a uniformly graduated scale, has a sufficient number of lines to mark positions accurately and permit interpolation. The reproduction of the iron spectra on the same plate in juxtaposition to the spectra of the unknown compensates for photographic and optical deviations which might make separate scales inaccurate. The positive identification of an impurity element, that is, qualitative analysis for traces, may require three or more lines of the element to determine that weak lines of the principal constituents are not coincident with these principal lines of the minor constituent. Tables and equivalent photographic charts (32) have been made available in recent years and provide the basis for rapid qualitative examination by spectroscopic means.

Emission spectra are easily produced for about 70 of 92 elements and provide a means for rapid qualitative analysis and the detection of minute impurity traces. The source used may be either arc or spark, depending on the nature of the spectra to be produced. The advantages of qualitative spectroscopic analysis are obvious in the ease of production and the absence of extended sample preparation or lengthy chemical treatments; however the sensitivity varies from element to element. In general, qualitative spectral analysis is not applied to nonmetals and gases because they require more modification. It has the advantage of detecting simultaneously rare elements that are not usually included in routine qualitative chemical analysis, such as indium, beryllium, vanadium, lithium, boron, and they are as easily detected as tin, lead, silver, or sodium.

Whereas qualitative analysis in its strict sense is the determination of only the presence of an element, such a limited approach is rare. Usually estimates are made of the amount of the element present and a semi-quantitative estimate of concentration can be made using persistent lines. Persistent lines are the last to disappear on dilution, and hence there is an order of intensity of lines which may be associated with the concentration. If the exposure and source intensity are controlled and the system calibrated for the matrix to be used, then the lines of the element under analysis can be classified into groups according to the concentration at which they are just barely observed. Lines can then be listed as indicative of 10, 1, 0.1, 0.01, 0.001, and 0.0001 per cent and several elements may be estimated simultaneously from the same spectrogram (5).

To extend the analysis to a quantitative determination, the intensity of the spectral line must be determined and correlated with the concentration of the element. The absolute intensity of the spectral line is not determined because there is much variation in intensity with the mode and conditions of source excitation. A study of the changes in the intensity of spectrum lines with change of element concentration and types of arc or spectra source of excitation shows that the excitation and concentration conditions produce a marked change in line intensities and that this change is not always equivalent in all lines. Therefore, to eliminate errors of measurement, a method known as the internal standard method was developed in which the intensity of the unknown line is determined relative to an internal standard line (33). Lines which are called homologous pairs are sought; they are lines which change proportionally with changes in conditions. Those pairs of lines which differ markedly from each other in their rate of change with a change of excitation conditions are known as control pairs. Control pairs and a third type of defining line, an exposure line, are used in defining and controlling the excitation conditions in an arc or spark (voltage, inductance, and capacity) and the exposure time. They ensure that the homologous pairs used in the analysis are sufficiently homologous to satisfy analytical requirements and that the intensities of the measured lines fall within the effective density range of the emulsion and photometer.

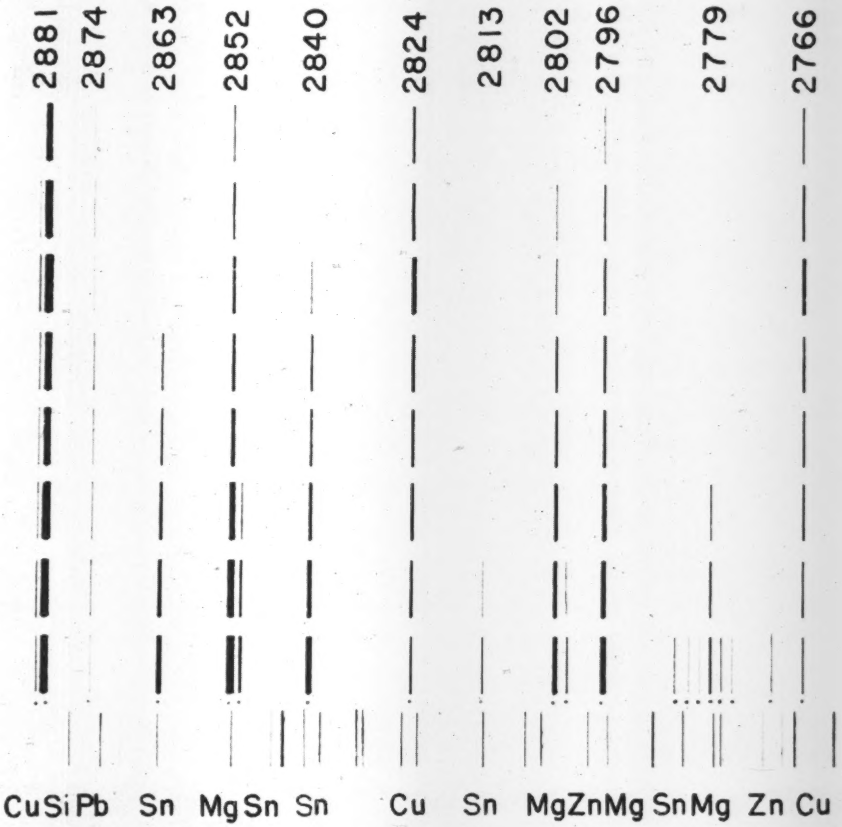


FIG. 16.—Spectrogram for Preparation of Densitometer Working Curve. The Variation in Spectral Line Intensity with Concentration Is Illustrated.

In the application of quantitative spectrographic analysis, an homologous pair of lines is chosen by experimentation or theoretical consideration, one line from the spectra of the unknown element and one from the internal standard element to form the homologous pair. The internal standard element may be an element that has been added to the sample in a known constant amount, or it may be the principal constituent of the sample

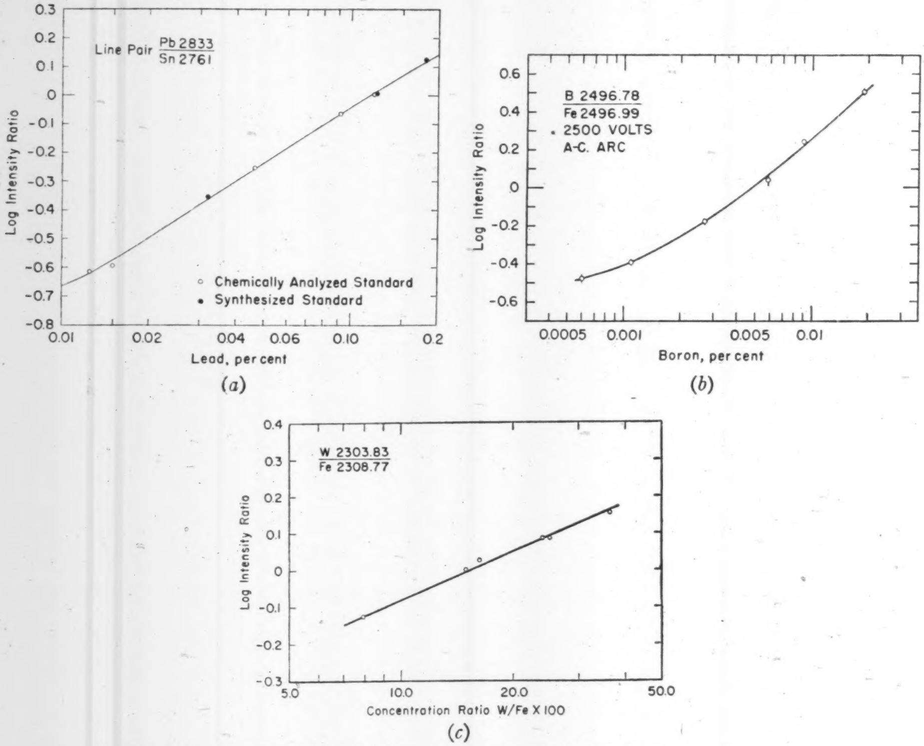


FIG. 17.—Examples of Working Curves for Emission Spectroscopy.

(*a*) Working curve of lead in tin alloy.

(*b*) Example of low concentration working curve; working curve of boron in steel. Note deviation from linear curve due to extraneous effects.

(*c*) Example of high concentration working curve; working curve of tungsten in iron. It is necessary to make an allowance for the reduction in iron concentration with the increase in tungsten concentration in order to obtain linear curve. *Courtesy B. F. Scribner.*

if this is relatively constant. In the latter case it is customary to use a weak line of the element in high concentration to compare with a strong line of the element in lower concentration, since more accurate comparison and less error due to observation and photographic deviation is obtained if the two lines to be compared are of nearly equal intensity.

A plot of the log of the relative intensity of an homologous pair of unknown and internal standard lines *versus* the log of the concentration of the unknown element has been found to be linear under ideal conditions and forms the basis of working or analytical curves (Fig. 17). It is essential that the relative intensity, if measured from the photo-

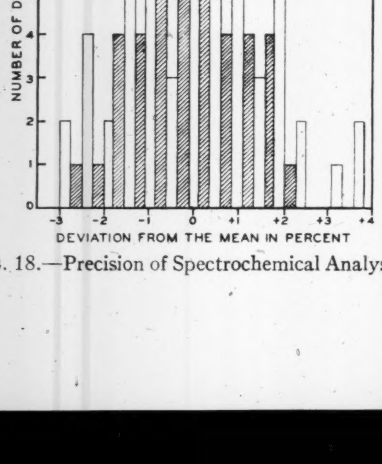


FIG. 18.—Precision of Spectrochemical Analyses.

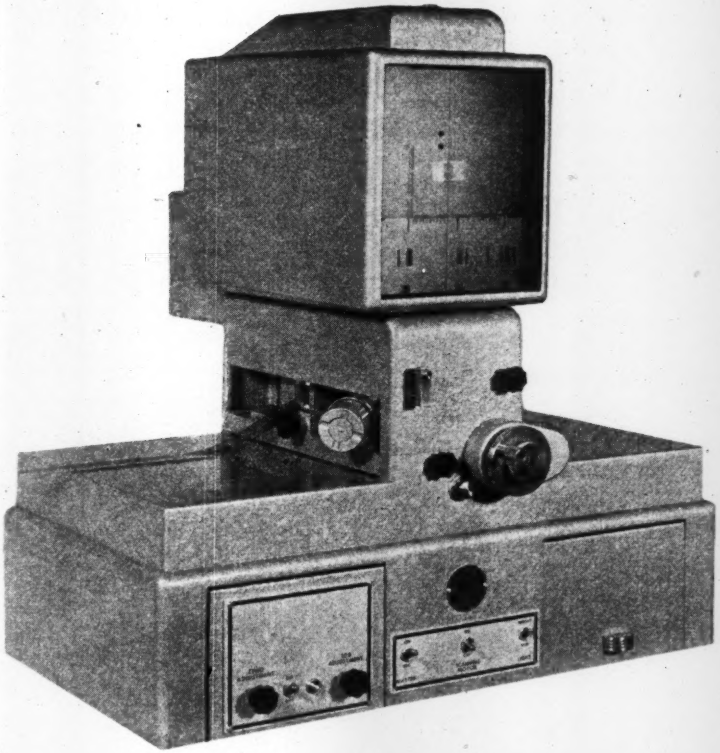


FIG. 19.—Film Projection Comparator Densitometer (Applied Research Laboratories).

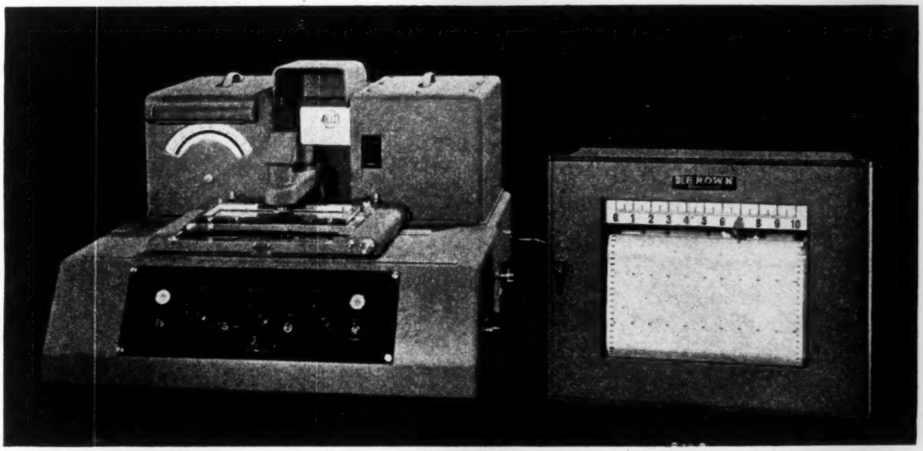

BROWN
0 1 2 3 4 5 6 7 8 9 10

FIG. 20.—Bausch & Lomb Photoelectric Densitometer.

graphic emulsion, be corrected for relative response of the emulsion and for background effects from stray light and continuous background due to excitation conditions.

Even with the correction it may be that the working curve is not linear, due to extraneous elemental effects (**34** to **36**), differing in excitation levels and self absorption or reversal. Hence it is often essential to project the working curve onto a nonlinear composition scale.

By suitable control of excitation conditions a reproducible scale can be produced to form the general basis of our quantitative procedures.

The accuracy of quantitative analysis is generally in the range of from 1 to 3 per cent of the amount present, and while such variation may not be as satisfactory as chemical analysis in the determination of high concentration, such as 30 or 40 per cent composition where a 1 per cent error might be expected, there is a distinct advantage in low concentration where a composition of 0.030 per cent could be easily confined within an error of less than 0.001 per cent quantitative instrumentation.

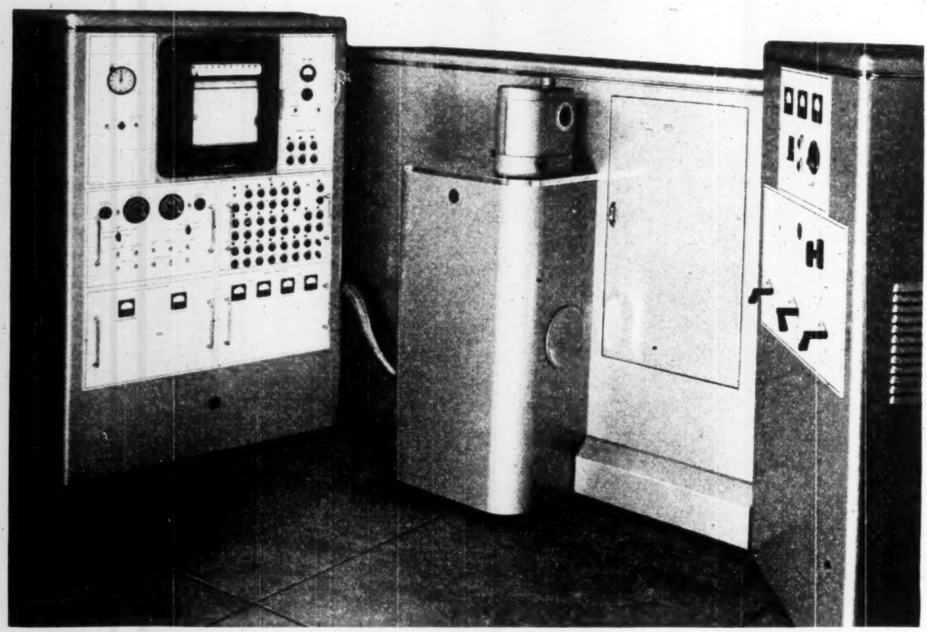


FIG. 21.—Production Control Quantometer. Spark-Arc Source Unit (Applied Research Laboratories).

Rapid and accurate quantitative analysis can be effected through the use of prism or grating spectrography and the measurement of the photographic densities by suitable densitometers (Figs. 19 and 20). These densities can be compared and corrected for emulsion characteristics through the use of calculating boards and projected slide rules. The range of application of quantitative analysis has been materially increased through the introduction of special "spectrum analysis" emulsions which give a nearly uniform density response or gamma on a wide spectral range (**37**).

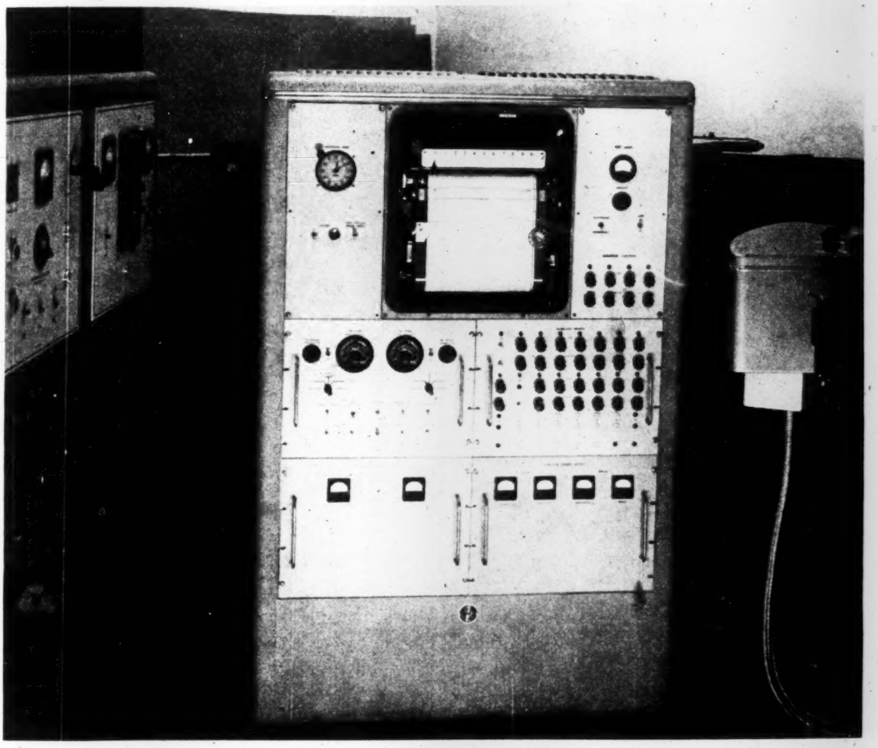


FIG. 22.—Direct Reading Installation (Applied Research Laboratories).

TABLE III.—COMPARISON OF REPEAT ANALYSES RESULTS OBTAINED WITH THE QUANTOMETER (SAMPLE 301) (**41**).

	Chromium	Nickel	Manganese	Silicon
Chemical Analysis	19.34	8.36	0.67	0.52
Spectrochemical Analysis:				
8:30 a.m.	19.30	8.39	0.68	0.51
9 a.m.	19.39	8.45	0.69	0.52
9:30 a.m.	19.38	8.44	0.68	0.51
10 a.m.	19.38	8.41	0.68	0.52
10:30 a.m.	19.40	8.31	0.67	0.53

Additional aids in the form of rapid photographic processing methods and recording microphotometers have made it possible to produce very rapid analysis of complex alloys or mixtures so that control changes or checks could be made during the processing of materials and corrections made in composition where necessary.

The success of quantitative spectrography by the photographic process has led to the development of photoelectric procedures of quantitative spectrographic analysis (**38** to **41**) in which the spectrum line is focused on a photocell surface and the intensity ratios of the homologous pairs determined by electrical balancing. The elimination of the photographic step in the process, while involving a considerably more complicated electrical instrumentation, has provided additional speed and accuracy which are economically justified where large numbers of routine analyses of a similar nature are to be made (Figs. 21 and 22).

The photographic and photoelectric methods have in some cases been combined (42) and conditions determine whether the method preferred will be photographic or photoelectric.

These control standards may be prepared synthetically or through careful chemical analysis within a laboratory, or advantage may be taken of their availability from laboratories which have prepared them for distribution. Their availability may in some laboratories remove the necessity of maintenance of a chemical laboratory control.

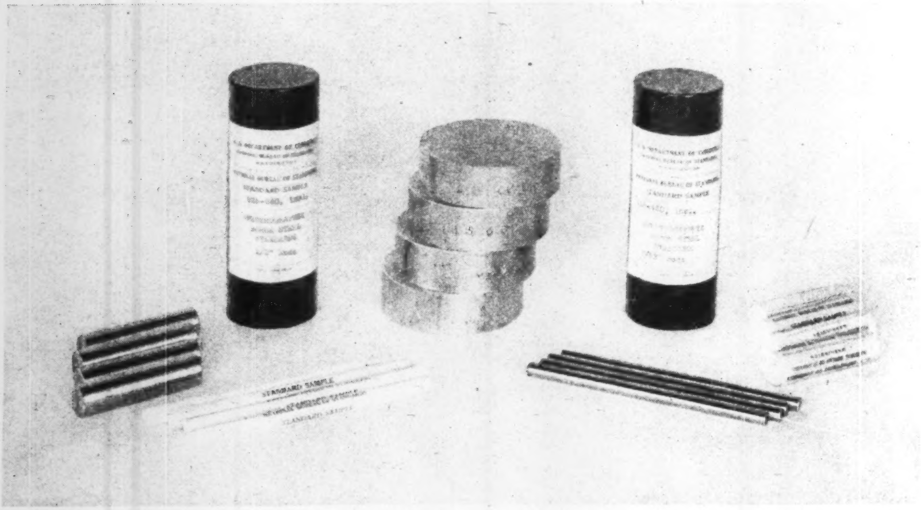


FIG. 23.—Standard Samples Issued by the National Bureau of Standards.

Control Standards:

The procedure of quantitative spectrographic analysis has been greatly facilitated by the availability of control standards of known composition. While working curves and selected homologous pairs may indicate the procedure to be used in an analysis, the excitation control, spectral transmission of the spectrograph, and the characteristics of the recording material are such that a separate working curve must be established for each instrument and condition used.

A.S.T.M. Committee E-2 on Emission Spectroscopy has for some time maintained a subcommittee on Standard Samples for Spectrographic Analysis and the published reports of this subcommittee give references to the source and composition of such standards (43). Many of these standard samples are maintained by the National Bureau of Standards as a part of its program of standard chemical samples (Fig. 23).

ABSORPTION SPECTROSCOPY

Although absorption spectra studies, especially with regard to quantitative determinations, were in general use before the development of quantitative emission analysis procedures, the methods of early investigators were either semi-quantitative in character or required long and tedious observations to arrive at acceptable quantitative results. Recently, with the development of photoelectric and electronic aids, the time of observation has been shortened to provide rapid and exact measurement.

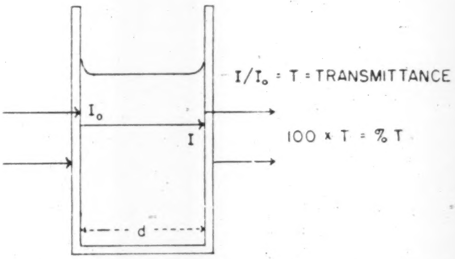


FIG. 24.—Graphical Indication of Transmission Terms.

Nomenclature:

The marked increase of interest in absorption spectra has emphasized the need of a more uniform and concise nomenclature system which takes into consideration the needs of the observers' technical use of the data as well as the physical principles involved. The methods and terminology used in the presentation of absorption spectra data are influenced by such factors as (*a*) precedent and previous usage, (*b*) the technical concepts involved in the data to be presented, (*c*) the notation prescribed by instrument producers and standard references, textbooks, or data forms, (*d*) the editorial policies and practices of technical journals and book publishers, and (*e*) the personal desires of the scientist or accords obtained by groups of research workers.

Transmission and Absorption:

Nearly a century ago Bunsen and Roscoe (**44**) outlined the general theory of absorption of radiant energy, which has usually been reproduced in what is known as the Bunsen absorption equation (Fig. 24): $I = I_o 10^{-kcd}$ where I and I_o represent the intensities of the transmitted and incident radiant energy, c the concentration of the absorbing material in a nonabsorbing media, d the thickness of the sample, and k the specific absorbance or the *Bunsen* or *extinction coefficient*. This equation may also be expressed as $kcd = \log_{10} I_o/I = 1/T$, where $T = I/I_o =$ transmittance and $T \times 100 =$ per cent T (per cent transmittance).

If the concentration is expressed as the molecular weight in grams (M), then the term k is replaced by ϵ (epsilon) and is known as the molar extinction $\epsilon = kM$.

Proposed System of Absorption Notation and Survey of Present Practice:

The following system of notation is proposed so as to define the various terms under discussion and permit an intercomparison of other methods of presentation (**45**):

Transmission:

I = Transmitted intensity of radiant energy.

I_o = Intensity of incident (or transmitted comparison) radiant energy.

T = Transmittance = I/I_o (Transmittancy is used to indicate relative transmittance if I_o is the transmitted comparison radiant energy).

t = Transmissivity, (T^{-cd}) is the Transmittance for a unit concentration and thickness.

Per cent T = per cent transmittance = $T \times 100$.

Absorption:

Absorption law: $I = I_o\, 10^{-kcd}$

A = Absorbance = kcd = $\log_{10} I_o/I$. This term (Absorbance) has been known as "Extinction" and represented by the symbol "E".

k = Absorptivity = A/cd = (specific absorbance or absorbance per unit concentration and thickness).

ϵ = Molar absorptivity = $k \cdot M$.

c = Concentration in grams per liter,

d = Thickness of cell in centimeters.

M = Molecular weight.

General:

The suffix -ion is used to indicate a process, as in the case of conduction or calibration; -or or -er to indicate a device as in generator or absorber; -ance to indicate a measurable quantity of a property such as resistance or conductance; and -ity to indicate a specific property of the measured substance such as density and conductivity.

The terminology recommended has been prepared with respect to the requirements of the principal users of absorption spectra data (chemists and biologists) and hence considerations of a technical or theoretical nature have in some cases been made secondary to those of simplification and ease of application. To avoid confusion, the proposed system represents a compromise between some of the present-day proposed systems, and the common usage and practice over a long period of time. In the notes to some of the terms there have been included some of the expressions of limited application for special application or reference.

Absorbance as a term and its symbol, A, are relatively new in absorption spectra notation and are the only real departure from the more accepted nomenclature of the past. The previous symbol and term for this has varied, although a preference would appear to be slightly in favor of extinction and E. To be consistent with the process of *absorption*, the name *absorbance* is preferred over extinctance. There is confusion in the literature as to the use of E for absorbance or molar absorptivity; the latter is usually indicated by the Greek letter epsilon ϵ, and this is often confused with the script E. In the gradual evolution through which our nomenclature is passing it is not unlikely that E may

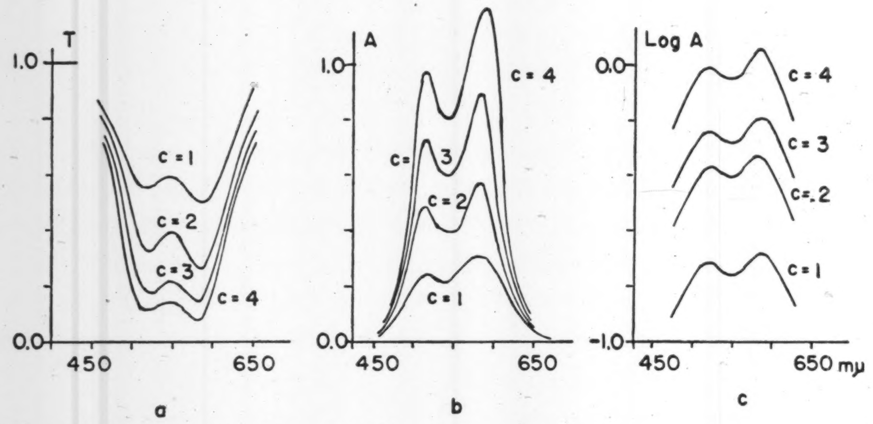


FIG. 25.—Comparison of (*a*) Transmittance, (*b*) Absorbance, and (*c*) Log Absorbance Presentation of Absorption Spectra Data.

replace ϵ as the symbol for molar absorptivity. There is still some question as to whether A should be called absorptance or absorbance, but the preference among spectroscopists seems to be in favor of absorbance (Fig. 25).

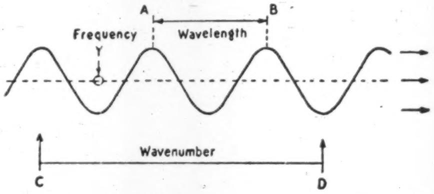
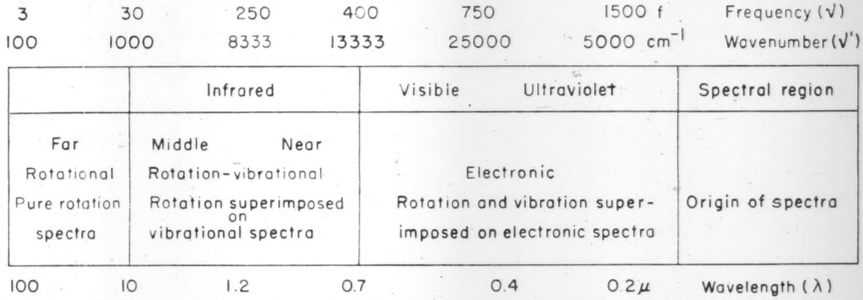


FIG. 26.—Graphical Indication of the Relation Between Wavelength (λ), Wavenumber (ν'), and Frequency (ν).
Wavelength (λ) = distance between A and B.
Wavenumber (ν') = number of waves in unit length (C-D).
Frequency (ν) = number of waves passing a fixed point (y) in a unit time

ϵ (*epsilon*) is almost universally used for molar absorptivity. A majority of the absorption spectra curves in recent chemical publications which characterize pure compounds, use epsilon as the ordinate. It is, however, given variously as ϵ, E, or E and to partially eliminate the confusion with the E as formerly used for *extinction* (now known as absorbance) it has been suggested that this latter use of E be discouraged. The symbol ϵ has been used consistently for a definite absorption property to a greater degree and extent than any other absorption designation (**45**). It should be recognized that the use of ϵ or log ϵ as ordinate is merely an indication that a molar quantity is involved in the calculated concentration and that the actual ordinate values are still in absorbance or log absorbance values. The use of log A and log ϵ-recording, while eliminating the additive character of the curves, provides a better comparison of weak and intense bands identification. The log A curves also possess a shape that is independent of concentration, which may be of assistance in the identification of pure compounds of unknown structure. Where A- or ϵ-values of considerable size are recorded and log A or log ϵ are not desirable forms to present the data on account of a desire to maintain the additive concentration notation, it is suggested that $A \times 10^{-2}$ or $\epsilon \times 10^{-2}$ be used.



	Infrared	Visible Ultraviolet	Spectral region
Far Rotational Pure rotation spectra	Middle Near Rotation-vibrational Rotation superimposed on vibrational spectra	Electronic Rotation and vibration superimposed on electronic spectra	Origin of spectra

FIG. 27.—Molecular States Producing Absorption in Various Radiant Energy Regions of the Spectrum.

Wavelength, Wavenumber, and Frequency:

From the basic theory of wave motion it is obvious that there are three methods of denoting wave characteristics: (*a*) the length of the wave (*wavelength* (λ)), (*b*) the number of waves or cycles in a unit distance (*wavenumber* (ν')), and (*c*) the number of waves or cycles passing a point in space in a unit time (*frequency* (ν)). The relation between these units may be expressed as

1/*wavelength* = *wavenumber* = *frequency*/speed of light

or

$$1/\lambda = \nu' = \nu/c.$$

A condensed arrangement of the radiant energy spectrum (Fig. 27) shows the relation of these three systems of notation and the principal spectral regions which are included in a general consideration of absorption spectra data presentation. It is to be noted that the spectral regions are not sharply defined and that the somewhat nebulous divisions are generally the limit of a source, transmitter, or receiver, rather than a marked change in radiant energy characteristics. In so far as absorption spectra effects are concerned, a more logical division would be between *vibrational*, *rotational*, and *electronic* spectra; however, the natural divisions which have been made are fortunate in approximating these subdivisions, so that no revision in subdivisions is necessary.

TABLE IV.—WAVELENGTH UNITS FOR RECORDING ABSORPTION SPECTRA.

Unit	Size of Unit	Range (from left to right)	Region
angstrom	meter × 10^{-19}	1000 to 1Å	Far or extreme-ultraviolet
mμ	meter × 10^{-9}	1500 to 100 mμ	Ultraviolet and visible
mμ	meter × 10^{-9}	400 to 750 mμ	Visible (color description)
μ	meter × 10^{-6}	1 to 200 μ	Infrared and far-infrared
cm.	meter × 10^{-2}	0.1 to 100 cm.	Microwave

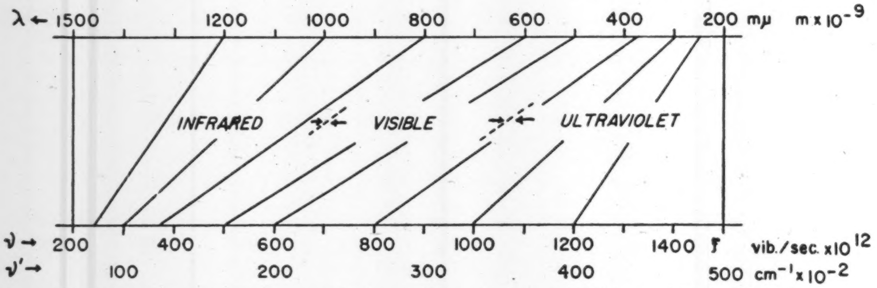


FIG. 28.—Wavelength-Frequency Relation in Near Infrared, Visible, and Ultraviolet Regions.

TABLE V.—FREQUENCY AND WAVENUMBER UNITS FOR RECORDING ABSORPTION SPECTRA.

Unit	Size of Unit	Range (from left to right)	Region
fresnel	Vibrations per sec. × 10^{-12}	100 to 1500f	Near-infrared, visual, and ultraviolet
megacycle	Vibrations per sec. × 10^{-6}	30 000 to 30 Mc	Microwave
(cm.$^{-1}$)	cm.$^{-1}$	400 to 5000 cm.$^{-1}$	Infrared
(cm.$^{-1}$ × 10^{-2})	cm.$^{-1}$ × 10^{-2}	100 to 500 cm.$^{-1}$ × 10^{-2}	Near infrared, visual, and ultraviolet

The term *near* as applied to *near-ultraviolet* or *near-infrared* should denote that portion of the indicated spectral region which is just beyond the limits of the visible spectrum. *Near-infrared* is generally accepted as that extension of the spectrum, beyond the red sensitive threshold of the eye, to which the photoemissive cell or photographic plate is sensitive. This includes approximately the region from 750 to 1500 mμ. The *near-ultraviolet* is that extension of the spectrum beyond the violet sensitive threshold of the eye which is transmitted by ordinary glass. This includes approximately the region from 400 to 300 mμ.

The term *far* as applied to ultraviolet and infrared spectra should denote the spectral region beyond the range of the usual recording or transmitting system. The *far-ultraviolet* region is the shorter wavelength region beyond the 200 mμ cut-off of the transmission of quartz and the limit of the usual gelatin photographic emulsion. The *far-infrared* region is the longer wavelength region beyond the 25 μ cut-off of the KBr prism and the start of the pure rotational spectral region.

Between these two fringe designations of near and far regions are the usual extended regions over which ultraviolet and infrared absorption spectra observations are made. It is recommended that these regions, 400 to 200 mμ in the ultraviolet and 0.75 to 25μ in the infrared be designated merely as the *ultraviolet* and *infrared* regions without a modifying prefix such as middle.

The *visible* region, like the above infrared and ultraviolet regions, is defined by the limits of the receiver—the human eye—and no sharp limiting value can be established. Individuals will vary with respect to their range of sensitivity, and some individuals, especially young blondes, can, when dark adapted, see as far as 350 mμ in the ultraviolet and to 900 mμ in the infrared, although the usually accepted limits of the visible spectrum are from 400 to 750 mμ. For color description it seems more desirable to use transmittance and wavelength for the spectral description; however, the apparent color of objects is influenced by the varied sensitivity of the eye, so that other systems of color description have been evolved which include this factor. These extended discussions and systems are beyond the scope of this paper (46 to 49).

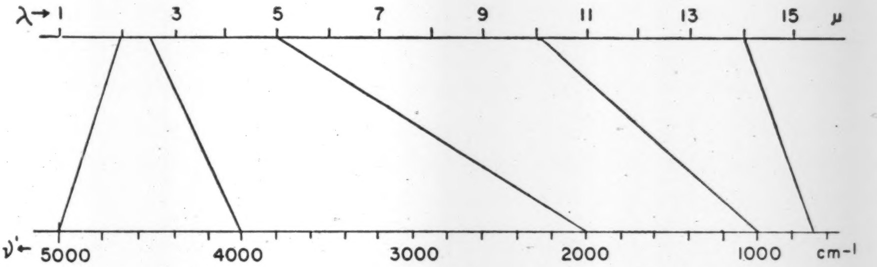


FIG. 29.—Wavelength-Frequency Relation in Infrared Region.

INFRARED SPECTROSCOPY

A logical subdivision of an absorption spectra discussion is to consider as separate subjects infrared absorption and visible and ultraviolet absorption. As has been indicated (Fig. 27) infrared absorption results from vibrational and rotational effects, and hence the chromophore type of polyatomic binding, as indicated in our subsequent discussion of the ultraviolet absorption spectra, does not produce multiple combination effects. The spectra produced are principally the inter-atomic rotational and vibrational spectra together with their overtone combinations.

Instruments:

The general principle of all infrared spectrometers embodies the usual components of source, cell, slit, focusing system, dispersing system (prisms or grating), and receiver element (Fig. 30). The radiant energy region to be studied will usually determine the character of these components. For the usual chemical analysis requirements, a lithium fluoride (LiF) or fluorite (CaF_2) prism is used for the region from 1 to 5μ, rock salt (NaCl) prisms are used for regions from 5 to 15μ, and potassium bromide (KBr) prisms are used for the regions from 15 to 25μ. Of these materials the rock salt (NaCl) prism is most widely used. Energy sources are usually radiant energy glowers such as the Nernst Glower or the Globar; cell windows are usually of the same material as the prism. Focusing is generally accomplished by a reflecting lens system, and receivers are usually thermopiles or thermocouples. More recently bolometers and thermistors have been used as detectors in infrared radiation incasement.

The evolution in absorption spectra instrumentation for infrared measurement (paralleling the change in the ultraviolet and visible instrumentation) has been the development of double-beam instruments (**50**, **51**) (Figs. 31 and 32). These double-beam instruments can incorporate in the final recorded graph a correction for variation in intensity of source over the observed radiant energy region, absorption due to solvent, and absorption due to the cell (Fig. 33). The ability to remove automatically these extraneous factors opens the way to accurate recording of concentration through absorption intensity and the more logical recording of infrared absorbance rather than transmittance (Figs. 34 and 35). There is a division of opinion among authorities as to the use of wavenumber in lieu of wavelength.

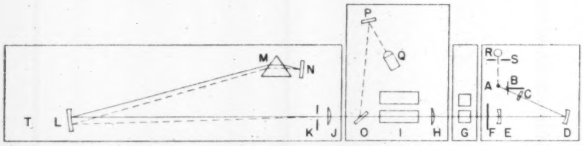


Fig. 30.—Optical Diagram of Beckman IR-2 Spectrophotometer.

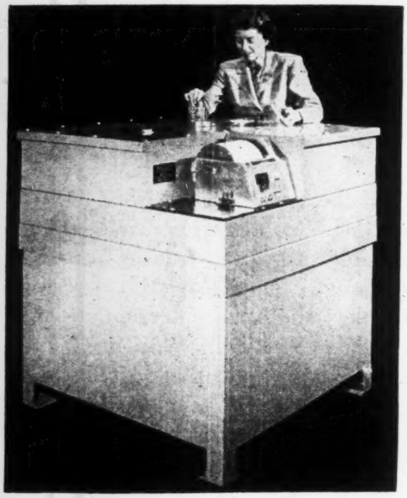
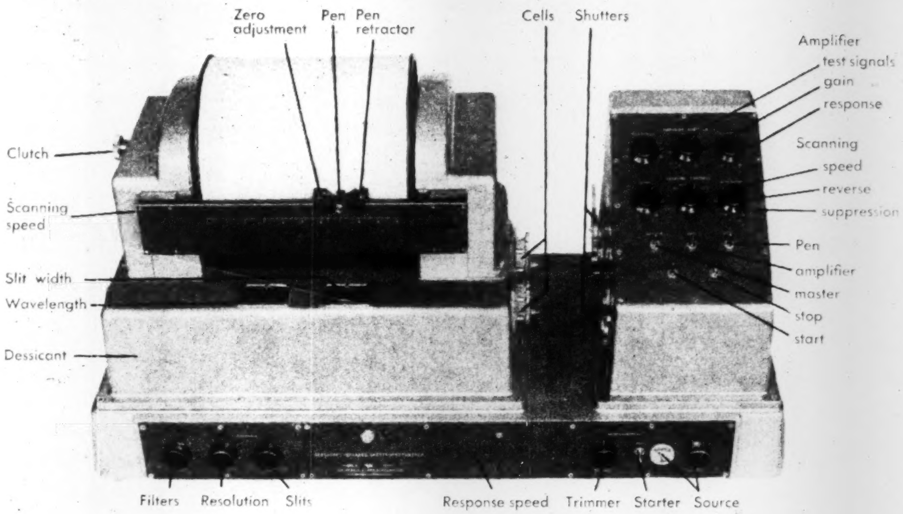


Fig. 31.—Baird Associates Recording Infrared Spectrophotometer.

The study of the absorption spectra of known substances has led to the assignment of certain frequencies as characteristic of certain atomic linkages (**52**). Tables of these frequencies have been prepared in both numerical and graphical form (Fig. 36) and from known spectral data (**53** to **55**) (Fig. 37) and from these tables it has been possible to predict the structural character of unknown samples.

A recent advance in infrared technology has been the incorporation of a


Zero adjustment
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Pen retractor
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Amplifier
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Clutch
Scanning speed
Slit width
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Filters
Resolution
Slits
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Trimmer
Starter
Source

FIG. 32.—Perkin-Elmer Recording Infrared Spectrophotometer (**51**).

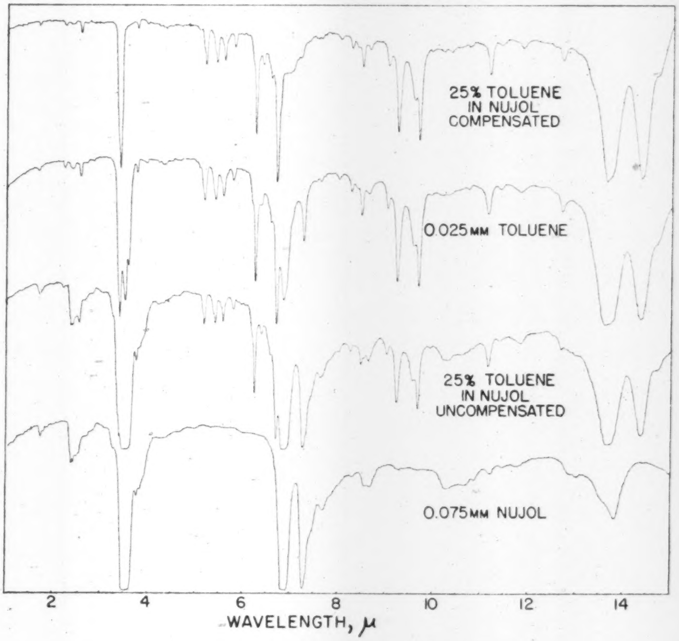

FIG. 33.—Compensation of Solvents.

0.5 μv. full scale 8 to 9 min. per spectrum (**51**)

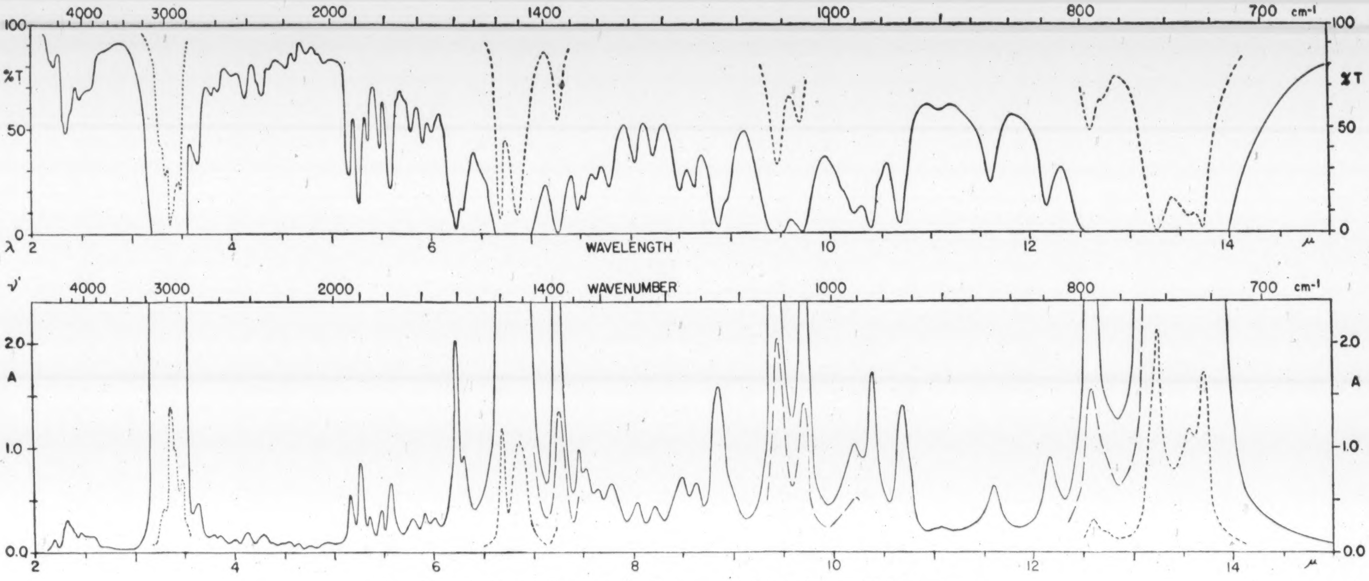

FIG. 34.—Infrared Absorption Spectra of 1-Methyl-2-Ethylbenzene.

(Data recalculated from NBS-API Project No. 44—Serial 349—Naval Research Laboratory.) Cell Thickness: ——— 0.250 cm.; — — — 0.125 cm.; - - - - 0.025 cm. Upper curve in percentage transmittance, *T*, lower curve in absorbance, *A*,; both curves plotted linear in wavelength.

micro cell for examination of single crystal material (Fig. 39) (56).

Quantitative analysis is possible in infrared applications, as is indicated in Fig. 40, in which it may be noted that the absorbance at 6.8μ of propane is directly proportional to the concentration (gas pressure) (51). Certified calibration standards are not generally available, although the test of resolution quality has often been the absorption spectra of ammonia, (Fig. 41). The best available compilation of data is that in the National Bureau of Standards—American Petroleum Institute series (53), and the collections by Barnes, *et al.* (54). Plans are in formation for the issuance of an extended bibliography of infrared data through the National Bureau of Standards—Ohio State Conference Committee. Some industrial consulting laboratories such as Philip

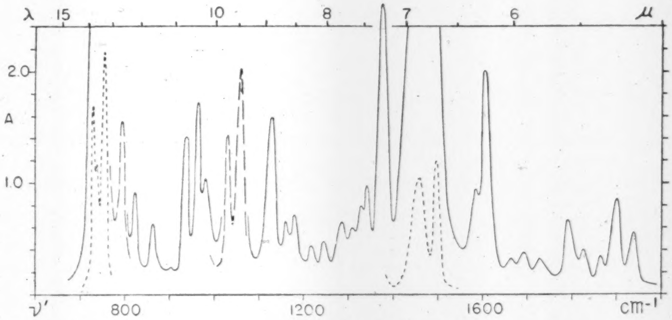

FIG. 35.—Infrared Absorption Spectra of 1-Methyl-2-Ethylbenzene (same as Fig. 34) Recorded in Absorbance and Linear in Wavenumber from 700 to 2000 cm.$^{-1}$ (15 to 5μ).

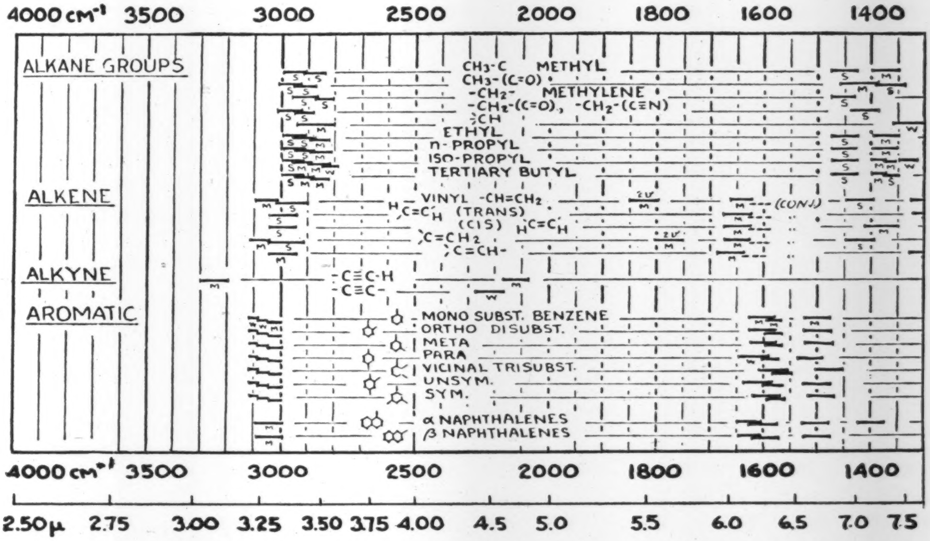

FIG. 36.—Portions of a Table Showing the Probable Positions of Characteristic Infrared Absorption Bands (52).

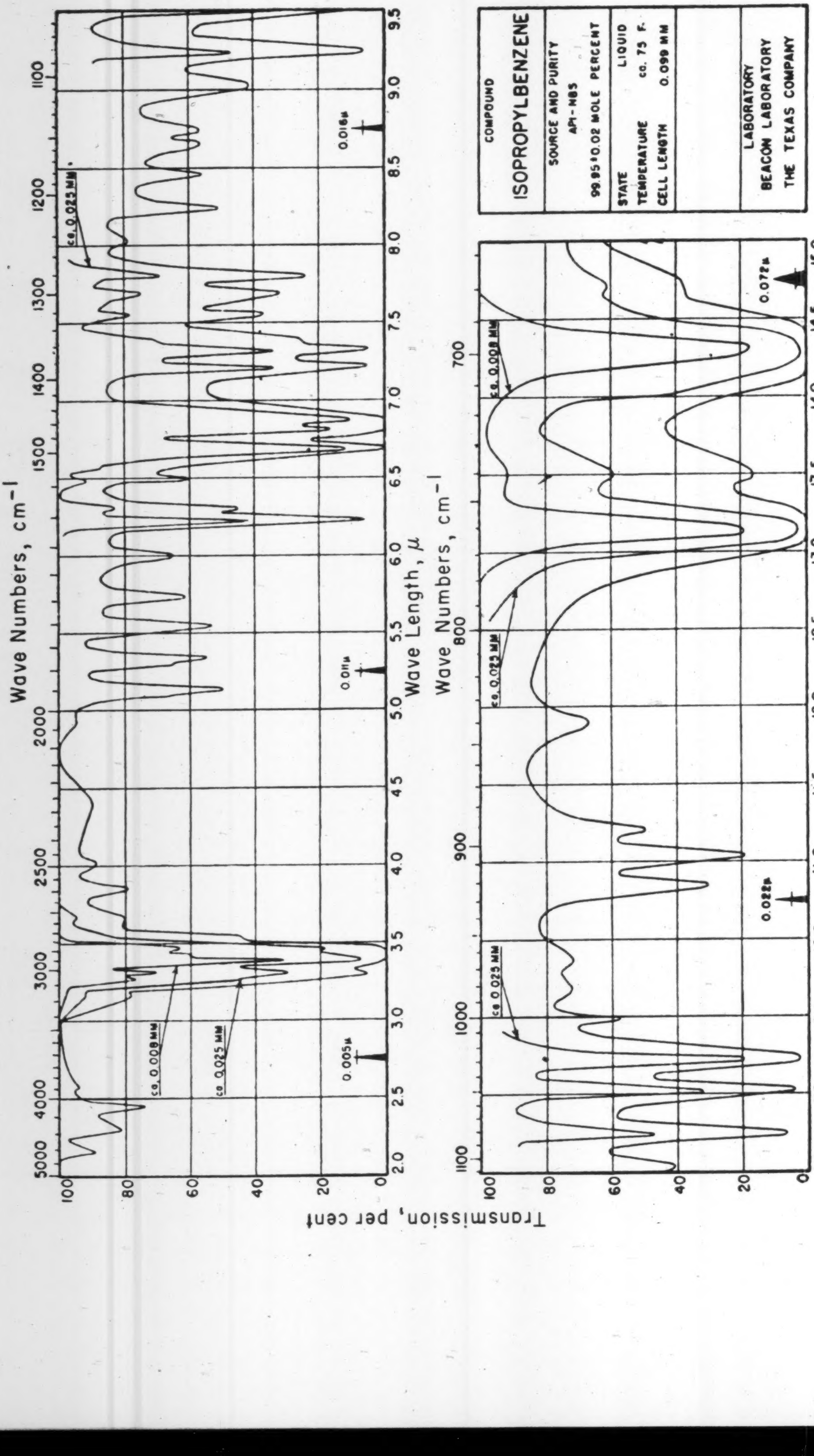

FIG. 37.—Typical Infrared Absorption Spectrogram used in NBS-API Project No. 44.

Sadtler offer subscription series of infrared data.

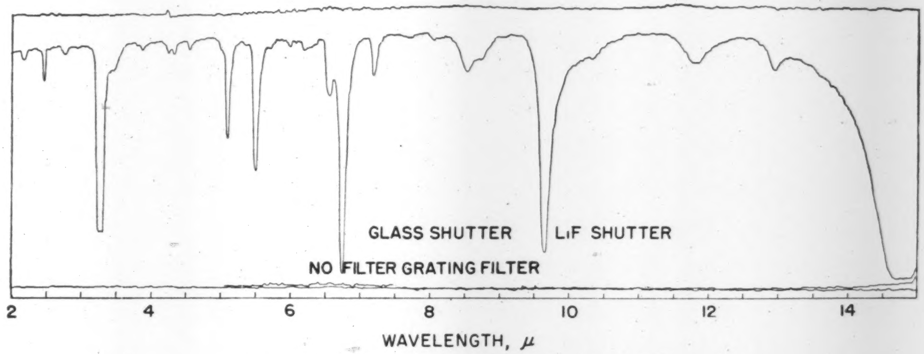

FIG. 38.—Infrared Spectrum of Benzene. (**51**).

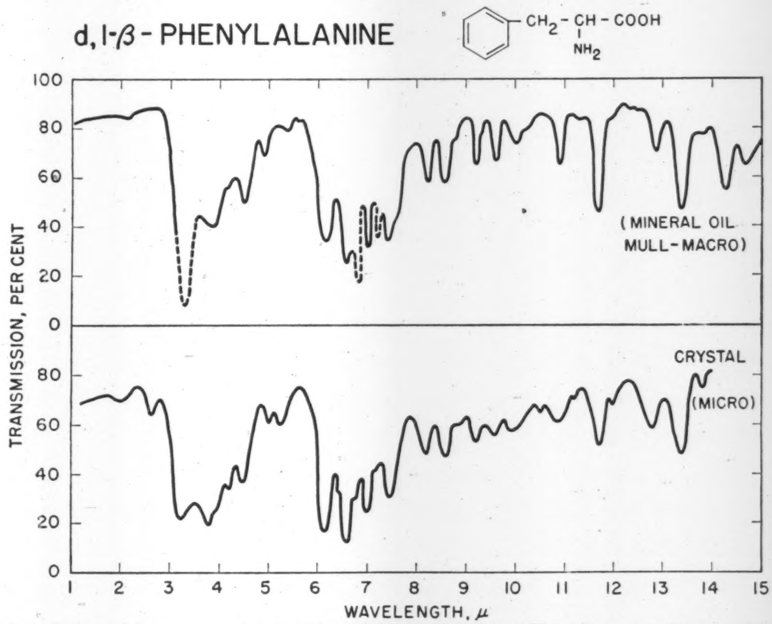

FIG. 39.—Infrared Spectrum of *d,l*-β-Phenylalanine in Crystalline State, as Obtained by Micro and Macro Techniques.

Dashed lines indicate absorption caused by mineral oil (**56**).

VISIBLE AND ULTRAVIOLET ABSORPTION SPECTRA

The electronic resonance effect with its superimposed vibrational and rotational effects is generally accepted as the explanation for absorption in visible and ultraviolet. The limits of these regions have been previously discussed. While visual color is the result of absorption in the visible region, the general subject of color and color description, which was the subject of a previous

Marburg Lecture by C. E. K. Mees (2), will be omitted from the present discussion.

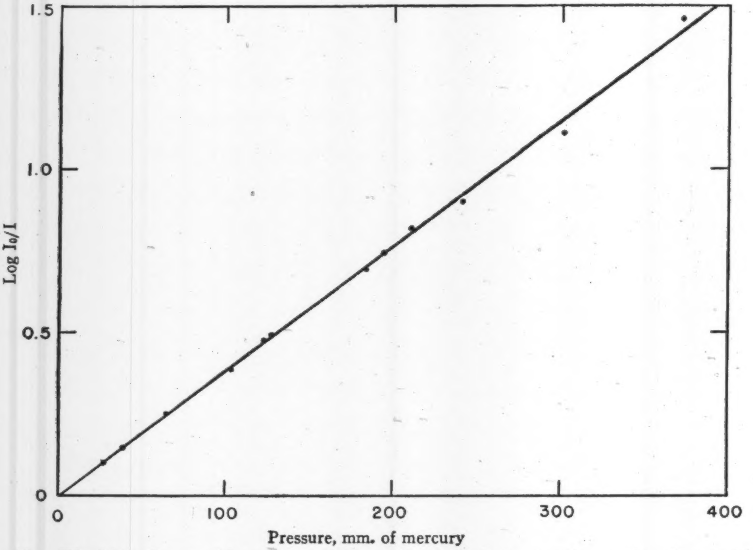

FIG. 40.—Working Curve. Linearity in Measuring 6.8μ Propane Absorption Bands.(51).

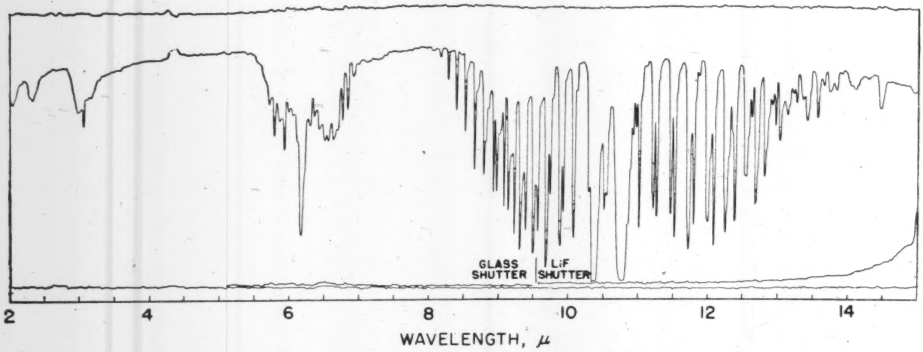

FIG. 41.—Infrared Spectrum of Ammonia.

Instrumentation:

Numerous double-beam or compensatory instruments have been developed over the years for both visual and photographic operation for the determination of visual and ultraviolet absorption. The distinct advance in recent years, however, has been in the application of photoelectric receivers and especially photomultiplier receivers to the detection and measurement of transmitted radiant energy (Fig. 42) and the adaptation of these detectors to automatic recording instruments which are associated with a wavelength or frequency drive (Fig. 43). An example of the resolving power of such a recording instrument is shown in its ability to record the vapor spectra of benzene (Fig. 44).

Descriptive Absorption Spectra:

Many of the terms used to describe infrared spectra can be used with equal correctness to describe visible and ultraviolet effects.

As a corollary to the ordinate and abscissa delineation of absorption spectra, it is desirable, especially for the applied deductions from the data, to present some of the terminology as may be useful in describing absorption spectra and the spectral changes which may result from variations in composition or conditions of observation. These descriptive terms have been classified as physical (optical) and chemical (structural), although a sharp separation is not possible and some of these effects belong in both classifications.

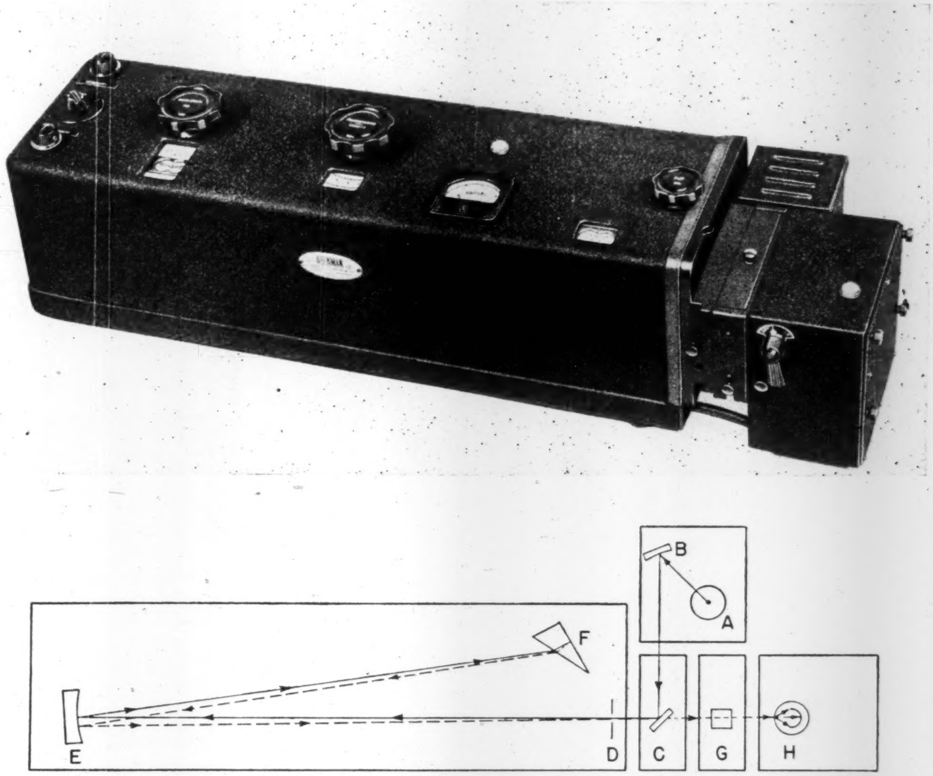

FIG. 42.—Beckman Photoelectric Spectrophotometer, Model DU.

Physical—Descriptive of the Observed Data

Position Change (λ or ν):

Bathochromic indicates a shift of the absorption band to longer wavelength values.

Hypsochromic indicates a shift of the absorption band to shorter wavelength values.

Color deepening is indicated by color changes resulting from a bathochromic effect, the order of color deepening being yellow-orange-red-purple-blue-green, and results from an absorption band shift from the ultraviolet, across the visible, and into the near infrared.

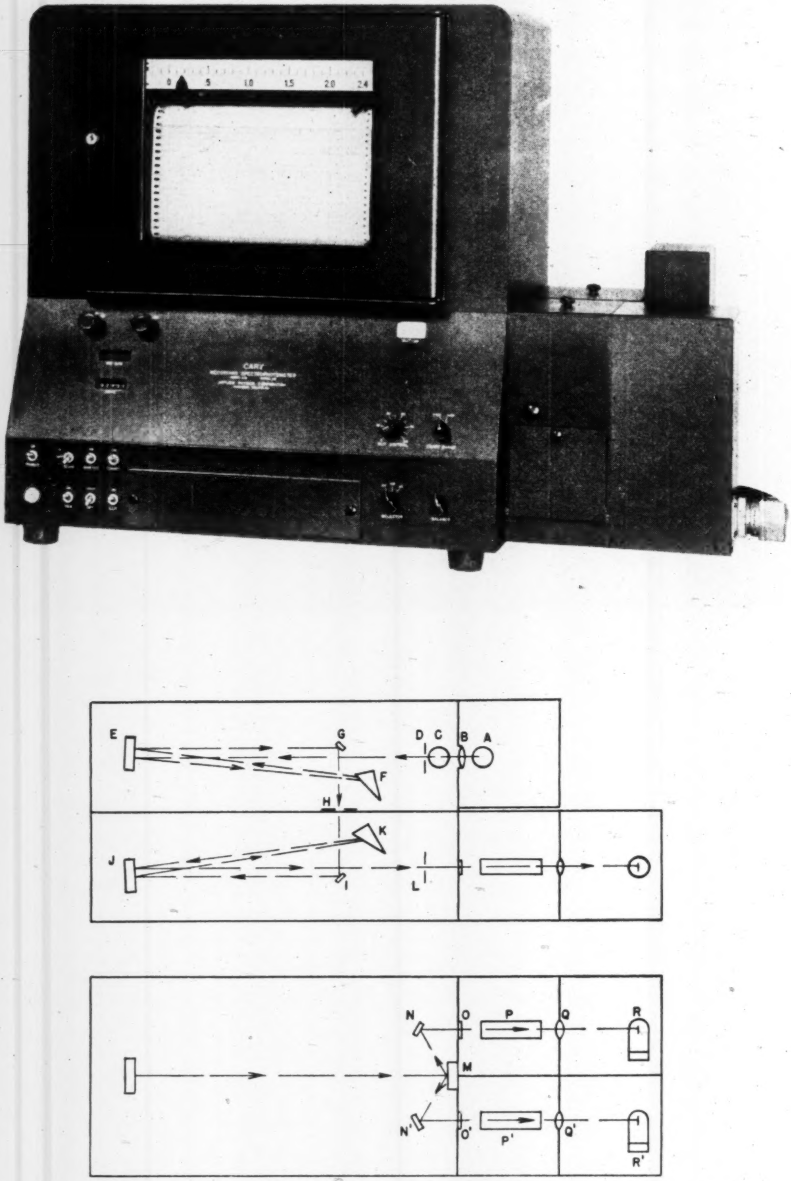
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FIG. 43.—Cary Automatic Recording Photoelectric Spectrophotometer (Applied Physics Corp.).

Lower half of figure shows the optical diagram of the Cary automatic recording photoelectric spectrophotometer.

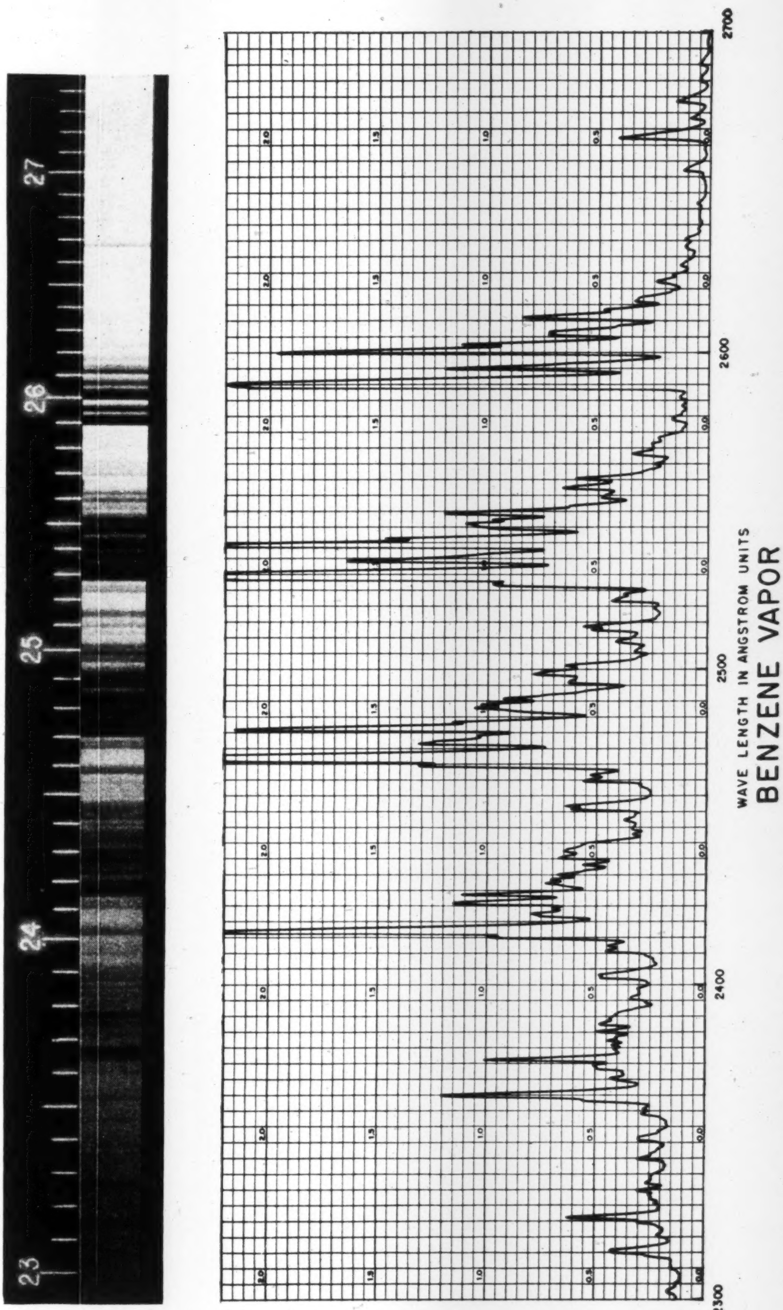


FIG. 44.—Spectra of Benzene Vapor. (*a*) Spectrum of Benzene Vapor. (*b*) Trace of Benzene Vapor Spectrum with Cary Automatic Recording Photoelectric Spectrophotometer.

Color lightening results from hypsochromic effects and is the reverse of color deepening.

Intensity Change (A):

Hyperchromic indicates an increase in absorbance.

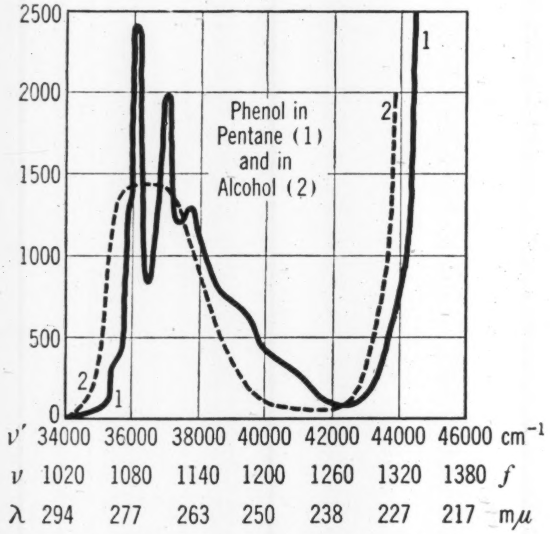


FIG. 45.—Absorption Spectrum of Phenol in Pentane (1) and Alcohol (2), Illustrating the Solvent Effect.

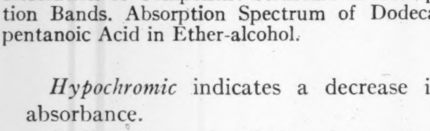


FIG. 46.—Influence of Temperature on the Resolution of Component Structure in Absorption Bands. Absorption Spectrum of Dodecapentanoic Acid in Ether-alcohol.

Hypochromic indicates a decrease in absorbance.

Fading is produced by destruction, removal, or conversion of absorbing material and may be merely a hypochromic effect or a combination of several effects.

Darkening is produced by a hyperchromic effect resulting from conversion of absorbing material.

Curve Shape and Structure:

Principal band generally refers to the first or strongest component in a series.

Symmetrical and *unsymmetrical* denote band shape. Narrow symmetrical bands generally indicate a single component, whereas unsymmetrical bands, especially with *inflections* or separate maxima, indicate *component structure*. *Resolution* of bands into component structure may in some cases be effected by improved instrumental techniques. Other procedures to improve resolution include *solvent effects,* such as the improved resolution in the spectra of phenol in pentane as compared with the spectra in alcohol, (Fig. 45) and *temperature effects* in which improved resolution is often shown in spectra observed at low temperatures (Fig. 46).

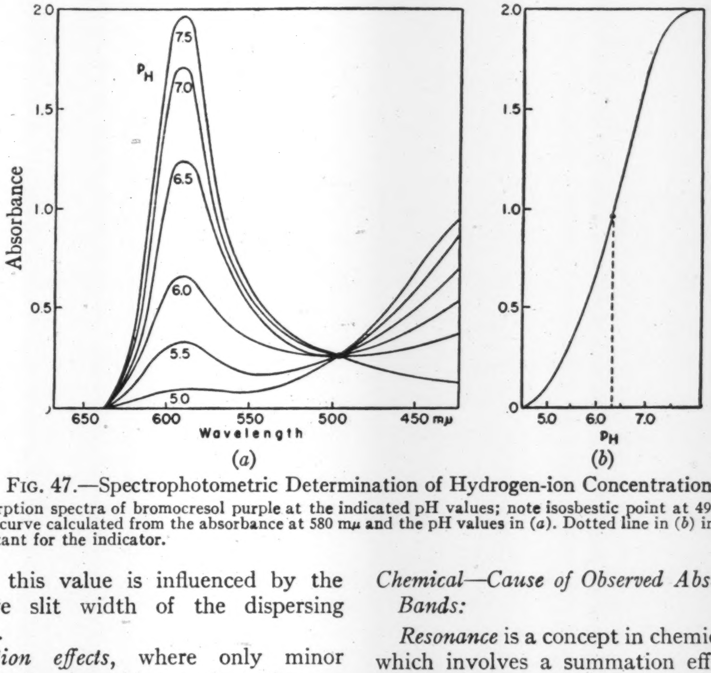
Absorption bands and component bands of a single compound which possess a simple numerical relationship on a frequency basis are said to belong to the same series. In infrared spectra these bands are known as *overtones* and in the visible and ultraviolet they are known as *harmonic* or *multiple series*. In both series the multiple relations are generally in simple whole numbers.

Half-band width is used to indicate the width of an absorption band at half its height. Such a description is applicable as a characteristic of the band only if the height of the band is expressed in absorbance units. In extremely narrow bands, this value is influenced by the effective slit width of the dispersing system.

Solution effects, where only minor shifts in band position or intensity are produced, are sometimes interpreted as physical rather than chemical effects and have been ascribed to changes in refractive indices, dielectric constant and other property variations of the solvent.

Isosbestic point is a point of equal extinction (absorbance) and is used to refer to the common crossing point of absorption spectra curves which results in the change of absorption spectra of a compound when the absorbing structure is changed, as in indicator changes and oxidation-reduction effects. The absorbance at such a point is characteristic of the amount of the compound present and is independent of the character or state of the equilibrium between the two absorbing substances (Fig. 47).

Dichroism is used to describe color changes which result from unsymmetrical absorption bands or unsymmetrical visual stimulation resulting from changes in the relative visual stimulation compared with the concentration change in the absorbing solution.



(*a*) (*b*)

FIG. 47.—Spectrophotometric Determination of Hydrogen-ion Concentration.

(*a*) Absorption spectra of bromocresol purple at the indicated pH values; note isosbestic point at 498 mμ. (*b*) The dissociation curve calculated from the absorbance at 580 mμ and the pH values in (*a*). Dotted line in (*b*) indicates dissociation constant for the indicator.

Chemical—Cause of Observed Absorption Bands:

Resonance is a concept in chemical-physics which involves a summation effect of dynamic equilibria between electromeric modifications and the resulting effect of the surrounding electronic environment. Single atoms may show rotational effects with resulting selective energy absorption known as *rotational spectra* which are evident in the far infrared. Bond effects between atoms result in selective absorption in the infrared known as *vibrational spectra*, on which are superimposed rotational spectra effects. Combination of bond effects result in electronic spectra which are apparent in the visual and ultraviolet regions and which in turn may have superimposed vibrational and rotational spectra effects. Vibrational spectra are classified according to bond characteristics and vector motion. Electronic spectra, excepting in the extreme or far ultraviolet are the result of molecular configuration involving characteristic combinations of unsaturated bonding which are generally known as chromophores.

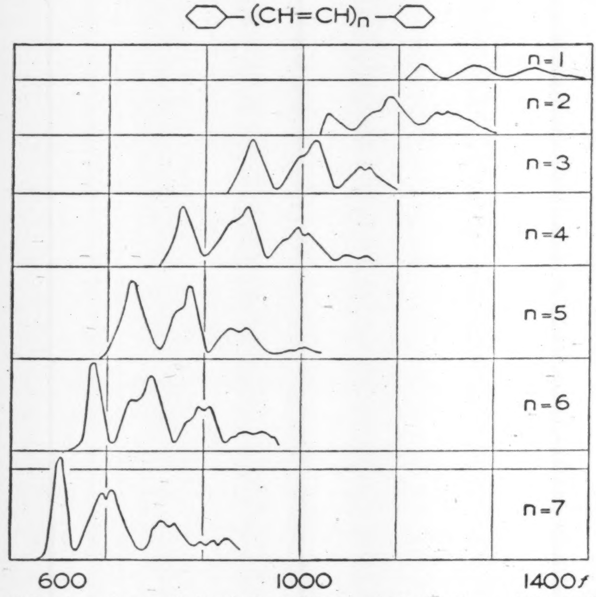
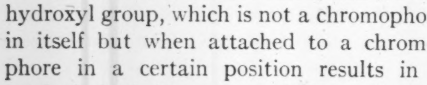


FIG. 48.—Effect of Conjugation in Polyethylene Diphenyl Derivatives, C_6H_5—(CH═CH)—C_6H_5.

Chromophores are radiant-energy absorbing groups such as



—N═N—, etc·

A *Chromogen* is that part of a complex molecule which contains a chromophore. An *auxochrome* is a substituent, such as the hydroxyl group, which is not a chromophore in itself but when attached to a chromophore in a certain position results in a marked bathochromic and hyperchromic effect. Simple chromophores do not as a rule show selective absorption in the visible spectra, but only in the ultraviolet region.

Combination of simple chromophores together may result in a larger chromophore which shows visual absorption.

There are three recognized modes of combination of chromophores:

1. *Separated*, in which the two chromophores, although in the same molecule, are separated by an insulating linkage, such as methylene (—CH_2—) or oxygen (—O—), so that resonance effects are not transmitted between chromophores, the resulting absorption spectra being the addition of the absorption spectra of the separate chromophoric radicals. There may be slight influences of one resonance structure on the other even though they are "insulated," and there are different degrees of insulation. In addition, increased molecular weight produces a small bathochromic effect.

2. *Conjugated*, in which the separate chromophores are combined together so as to permit resonance transmission and to produce a single larger chromophore. The effect on the absorption band is almost multiplying, rather than additive, in that both bathochromic and hyperchromic shifts are produced. There is a limit to the number of unsaturated or resonance centers that

may be conjugated together, so that unstable compounds or divided resonance may result from over-conjugation. In addition to direct attachment together of chromophores, it is possible to combine them together by transmitting linkages such as the ethenylene linkage (—CH=CH—) (Fig. 48). Covalent and coordinate linkages are capable of transmitting effects, whereas ionic linkages produce insulating chromophoric effects. Combination of simple chromophores together by conjugated linkages may produce larger chromophores which possess unusual stability or color properties and to which distinctive names such as benzenoid, quinoid, indigoid, flavanoid, etc., are given to simplify nomenclature and discussion.

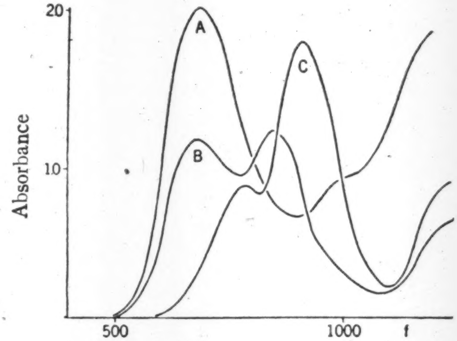


FIG. 49.—Conjugated and Cumulative Chromophores.

Absorption spectra of disazo dyes, (A) with the two chromophores conjugated, (B) with the two chromophores cumulatively linked, and (C) with the two chromophores separated.

3. *Cumulated,* in which the two resonance centers share electrons in common so as to permit only one of the two forms to exist simultaneously, as in allene ($CH_2=C=CH_2$), and certain bisazo dyes. There is usually not a complete suppression of resonant forms and, while a hypochromic effect is apparent, there is usually some bathochromic effect as well as the apparent single chromophore (Fig. 49).

Chemical or structural changes in absorption bands involve changes from one chromophoric form to another or the destruction or formation of a chromophoric form. *Indicators* generally imply change from one color form to another as a result of a change in acid or alkaline condition. A single indicator may show more than one change in color over a range of pH conditions. A study of the absorption spectra as the color changes shows that there is an equilibrium between the two structural forms and an isosbestic point of common absorbance can be determined. The absorption changes of indicators do not have to be visual and may involve changes in ultraviolet absorption. They may also be produced by oxidation-reduction, ionization, hydration, and other reagent effects.

If the reaction results in a visual colorless form in one modification, this colorless form is known as the leuco form. Production of colored forms from the leuco form is often referred to as color development. A compound is considered to be *thermochromic* if a reversible absorption change is produced by heating or cooling. *Photochromic* and *radiochromic* compounds show reversible absorption effects resulting from radiant energy, X-ray, or radioactive exposure.

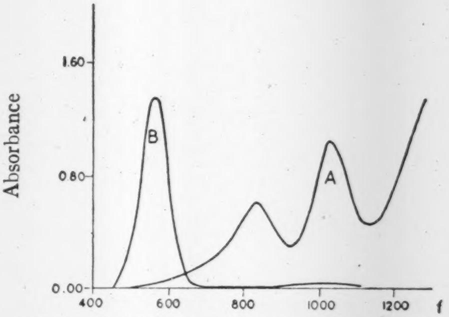


FIG. 50.—Absorption Spectra of Iodine in Alcohol (A) and Carbon Tetrachloride (B).

Chemical *solvent effects* such as solvation are common and must be considered in the choice and purity of solvents. For example, iodine in carbon tetrachloride gives a purple solution with a sharp absorption band at 540 mμ. The presence of a small amount of alcohol in the carbon tetrachloride results in a brown color with an absorption maximum in the ultraviolet. A study of this effect shows that the brown color is essentially the absorption spectra of iodine in alcohol, in which it forms a complex, and that the carbon tetrachloride-alcohol solvent is actually an alcohol solution of the iodine which is merely diluted by the carbon tetrachloride as an inert diluent (Fig. 50).

Variation in molecular weight, when such a change is involved in the resonance structure or portion of the molecule, results in a shift of the absorption band to a longer wavelength (lower frequency) as the mass is increased (Fig. 51).

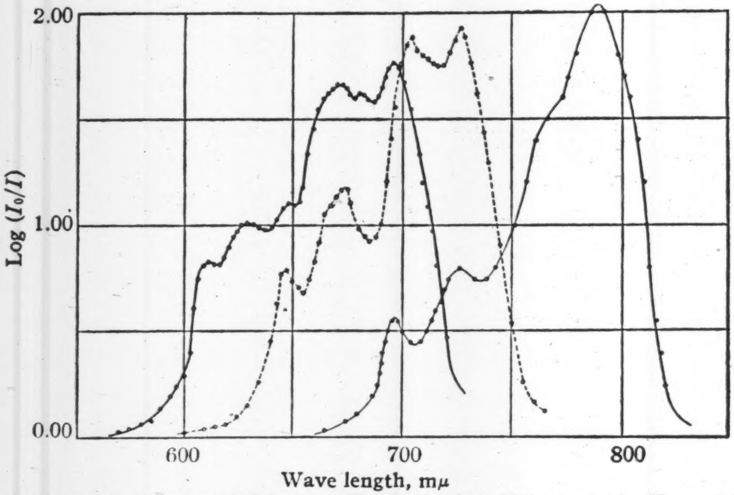


Fig. 51.—Absorption Spectra of Cobaltous Halides in their Halogen Acids: Heavy Line, $CoCl_2$ n HCl; Dotted Line, $CoBr_2$ in HBr; Light line, CoI_2 in HI.

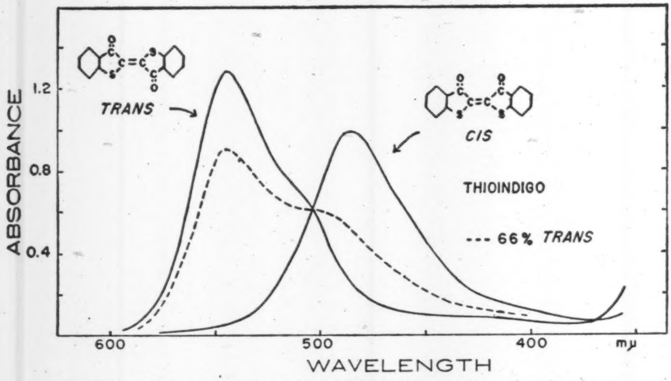


Fig. 52.—Absorption Spectra of Thioindigo, *cis* and *trans* forms.

Geometrical isomerism of *cis* and *trans* forms is demonstrated by their difference in absorption. An interesting example of such in what might be termed a "chameleon" effect has recently been demonstrated (57) (Fig. 52) in thioindigo. A blue solution (*trans*) with an absorption band in the red, if irradiated with light of the wavelength of its absorption (red light), is converted to a red solution (*cis*), which has an absorption band in the blue. The reaction is reversed if the red (*cis*) solution is irradiated with light of the wavelength of its absorption band (blue light) so as to produce a blue solution (*trans*).

Structural isomerism is in many cases more easily detected in the ultraviolet and visible than in the infrared absorption spectra. This is especially true when conjugation plays an important part in the resonance structure. In Fig. 53 it will be noted that blocking of the available electrons on the carbon adjacent to the aromatic ring by substitution with methyl groups results in a spectra of tert-butyl benzene which is not far different in average contour from benzene, whereas the normal chain isomer *n*-butyl benzene is quite similar to toluene in which the unsymmetrical molecule shows a marked shift of intensity to the longer wavelength component.

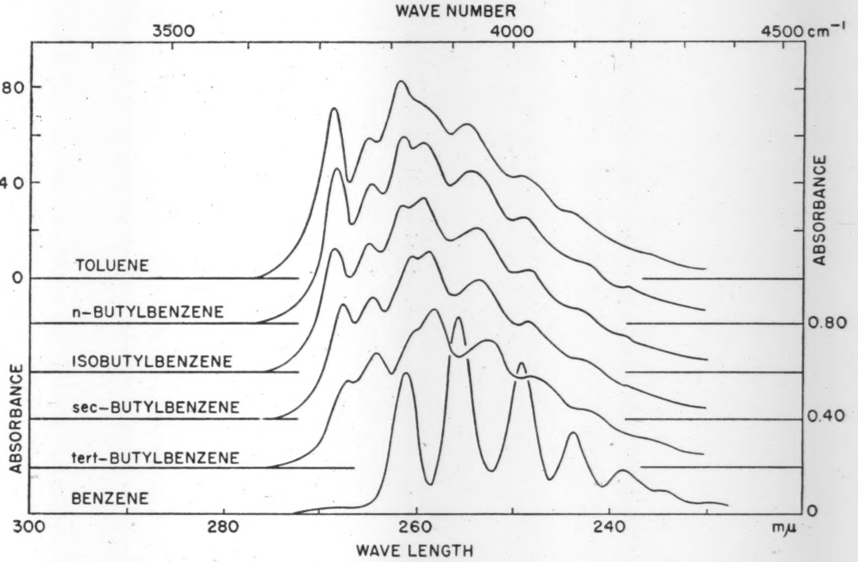


FIG. 53.—Absorption Spectra of Toluene, Benzene, and the Butyl Benzenes.

Two other examples, involving azo dyes, should suffice to show the possibilities of the use of absorption spectra to explain structural variation in complex resonance forms. In the separation of the azo chromophores in a disazo compound it is possible to insulate completely the two chromophores so that they act as separate molecules (Fig. 54). The degree of insulation may be reduced by closer coupling or coupling through a resonance transmitting linkage so as to produce eventually a single resonator.

In the benzidine series (Fig. 55), it is further possible to insulate the two azo chromophores by forcing the benzidine rings out of plane by introduction of methyl groups *ortho* to the connecting link between the two phenyl rings and in a reverse manner the phenyl rings may be directed towards an increase in their apparent coplanar form by substitution of a methoxy group in the meta position.

Quantitative Analysis:

In addition to qualitative identification which is obviously possible from the finger-print type of spectra which many compounds yield (Fig. 56), it is also possible to estimate the amount of many substances present in mixtures. For example mixtures of chlorophyll A and B can be analyzed by a measure of the absorption band from 420 to 480

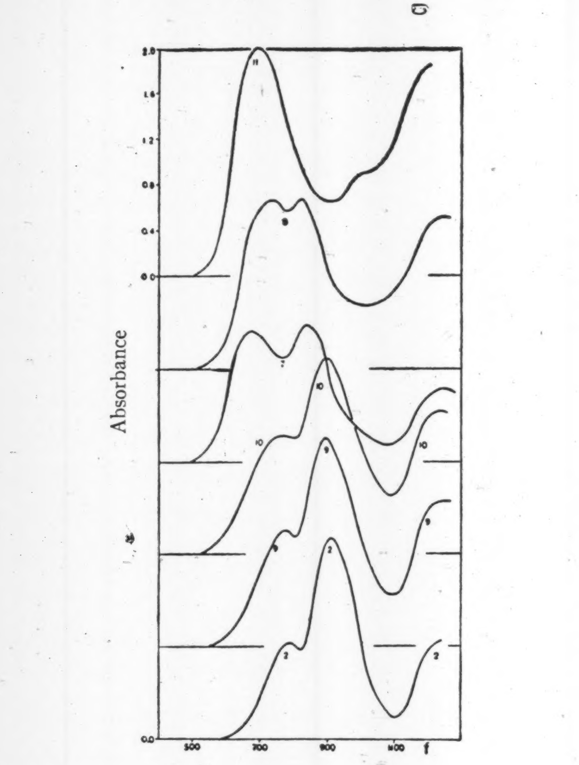
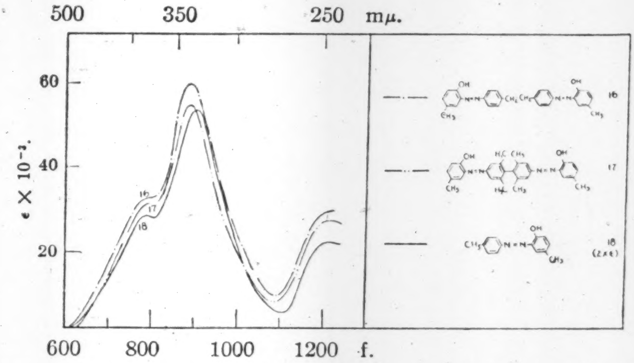
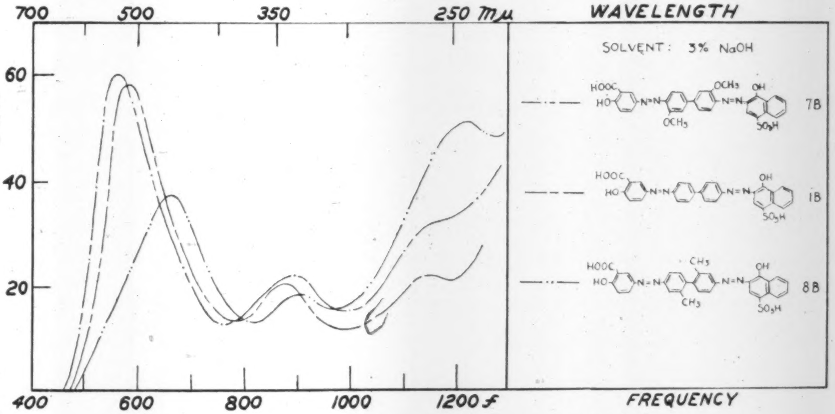


FIG. 54.—Absorption Spectra of Chromophores Linked by Conjugated and Non-conjugated Groups.



(*a*) Absorption spectra in alcohol solution of two disazo dyes showing restriction to molecular conjugation and a monoazo dye of suitable construction to represent most nearly a half-structure of these molecules.



(*b*) Absorption spectra of three unsymmetrical disazo dyes in basic media.

FIG. 55.—Absorption Spectra of Disazo Dyes in Alcohol and in Basic Media.

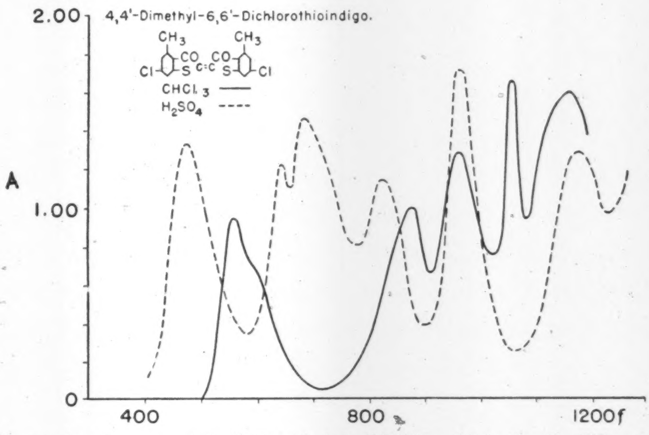


FIG. 56.—Absorption Spectra of 4, 4'-dimethyl-6, 6'dichlorothioindigo in Chloroform (———) and Sulfuric Acid (- - - -) Solution.

mμ. In research and commercial practice, many dyes, drugs, and other chemicals with resonance absorption are qualitatively identified and quantitatively estimated by their absorption spectra. Standard methods of analysis for vitamin A involve the use of its absorption band at 328 mμ.

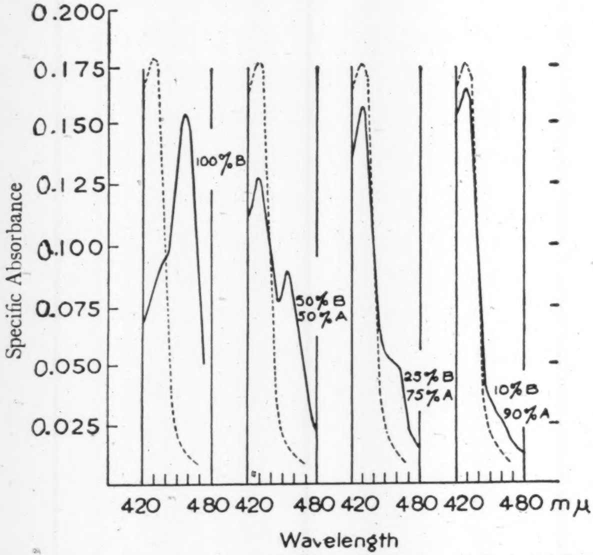


FIG. 57.—Absorption Spectra of Mixture of Chlorophylls A and B (solid line) and Chlorophyll A (dotted line).

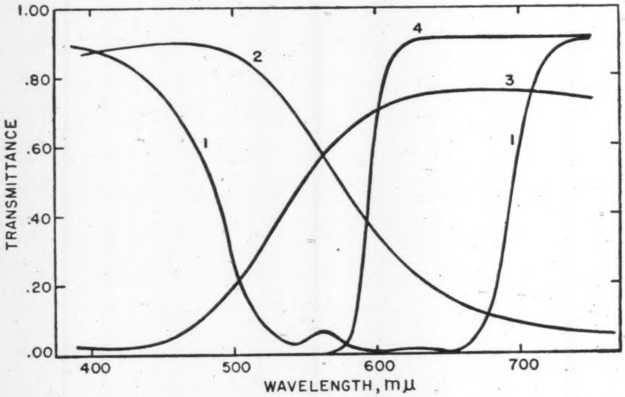


FIG. 58.—Typical Spectral Transmittance Curves of Glass Standards.

STANDARDS

Standards are essential for calibration of equipment and assurance as to the intercomparability of data observed on different instruments or from different laboratories. In the infrared, ammonia has proven useful as a test compound to test the resolution (Fig. 41). Benzene provides a useful wavelength calibration (Fig. 44 (*b*)). In the visual range, colored glasses are often used to calibrate transmittance (Fig. 58). These standards which are issued by the National Bureau of Standards are quite satisfactory for transmittance scale calibrations but are not as satisfactory for absorbance. For this latter purpose, a different set of standards (Fig. 59) should be used which would provide plateaus of maxima or minima at various absorbance values over the range measured and which would not be appreciably affected by

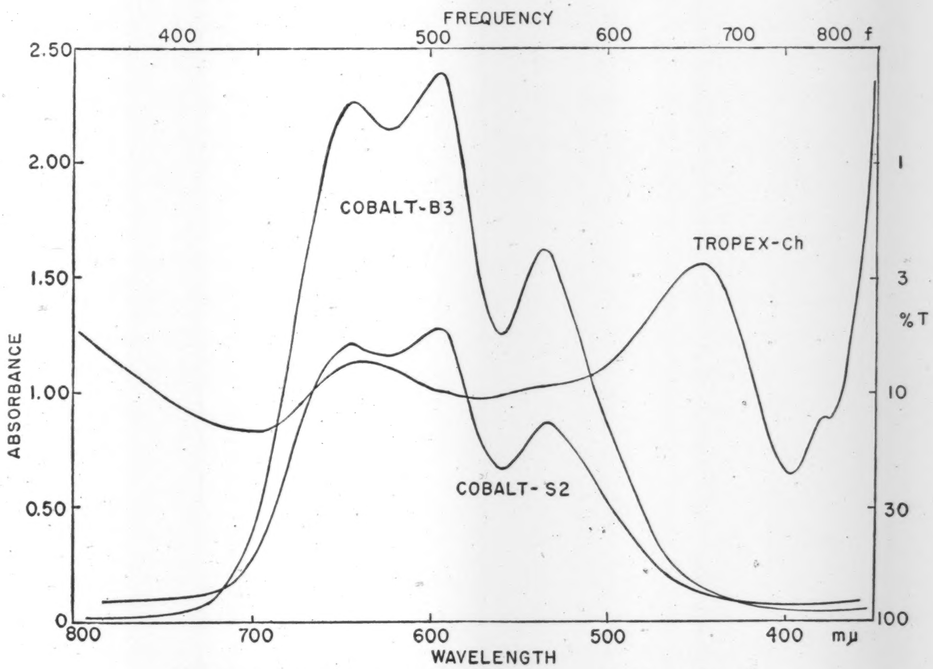


FIG. 59.—Typical Spectral Absorbance Curves of Colored Glasses.

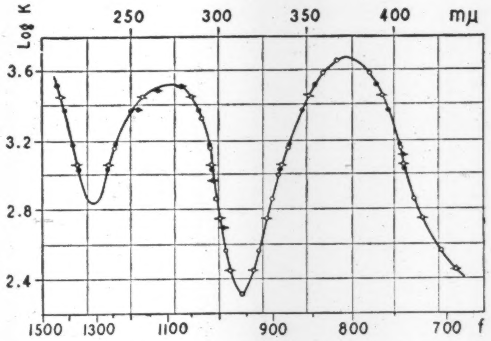


FIG. 60.—Absorption Spectrum of Potassium Chromate. Molar Solution in 0.05 *N* KOH. *d* = 1 cm.; • Sector Method; –•– Photoelectric Method; ○ Arc Screen- –○– Spark Screen (**5**).

slight deviations of the wavelength setting. Wavelength values, on the other hand, are best checked by the use of a mercury source. For ultraviolet absorbance calibration, solutions of potassium chromate (Fig. 60), potassium nitrate, or potassium acid phthalate have been recommended.

Resolution tests can be made with rare earth (didymium) glasses, or benzene vapor (Fig. 44 (*b*)).

REFERENCE DATA

National Bureau of Standards—American Petroleum Institute curves of the visible and ultraviolet absorption spectra of hydrocarbons and related substances have been issued on a large number of substances. A new National Bureau of Standards series on other types of substances is in the process of initiation and other compilations have been assembled (58, 59).

The wide distribution and availability of spectrophotometric equipment (for example, over 3000 Beckman DU instruments are now in use in various types of laboratories throughout the world), has resulted in a marked increase in absorption spectra data and application as a part of the fundamental constants and measurements used to describe and characterize new compounds and reaction states. In the past two years, about 10 per cent of all of the technical papers in chemistry have included some absorption spectra data. In the *Journal of the American Chemical Society*, of the approximately 2500 papers published in the past two years, over 300 papers included absorption spectra data.

With the improvement and development of rapid automatic and photoelectric instruments in the visual and ultraviolet regions (Cary, Beckman, General Electric, Brode, Harrison, Gibson, etc.) and double-beam automatic instruments in the infrared (Baird, Perkin-Elmer, Hilger, Beckman, etc.), it can be expected that the next decade will witness an even greater emphasis on the publication of absorption spectra data.

Just as the medical research worker, as an applied microscopist, has learned to use microscopic equipment without a comprehensive knowledge of the field of optics, so it has been my contention that chemists and engineers learn to use spectrographic equipment in chemical analysis without a background of extensive physical theory. This concept of applied spectroscopy has formed the basis of textbooks and courses in many of our leading universities and the resulting trained technologists are now using these techniques in the fields of testing and research.

Acknowledgment:

I feel that I should not close this discussion without some expression of appreciation on the part of spectroscopists to A.S.T.M. for its constant and effective support of this new field.

In 1932, under H. V. Churchill of the Aluminum Research Laboratories, Committee E-2 on Emission Spectroscopy was organized. Mr. Churchill served as Chairman from 1932 to 1946 and was succeeded by Bourdon F. Scribner of the National Bureau of Standards. Secretaries of the committee have included C. C. Nitchie, B. F. Scribner, and Mary E. Warga.

This committee has held some three symposia at national meetings of the Society, has published survey work on standard samples (Brode—Scribner—Corliss) (43); and has published three editions of an emission spectra bibliography by Meggers and Scribner) (60). In the 1946 Book of A.S.T.M. Methods of Chemical Analysis of Metals (61), 5 tentative methods of spectroscopic analysis were published and in this year's meeting it is expected that an additional "volume" of some 54 suggested methods for emission spectrochemical analysis will be approved.

The recent organization of Committee E-13 on Absorption Spectroscopy will, it is hoped, lead to an equally effective service in this field.

With the cooperation of the National Bureau of Standards, and the scientific organizations as well as industry and universities we may look forward to advances in the provision of standard samples, standard data catalogs, standard methods of analysis, and improved techniques and equipment in this field.

Acknowledgment is made to John Wiley and Sons for permission to reproduce certain figures from "Chemical Spectroscopy" (5) written by the author and from the Academic Press for reproduction of figures from the chapter written by the author in Berl's "Physical Methods of Chemical Analysis" (4).

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THE INFLUENCE OF STRAIN RATE ON SOME TENSILE PROPERTIES OF STEEL*

By D. S. Clark[1] and P. E. Duwez[1]

Synopsis

The inability of impact tests to reveal the true influence of strain rate on the tensile properties is demonstrated. The fundamental requirements of a device by which strain rate effects can be studied are considered and the details of a machine that has been constructed are presented. The results of static and strain rate tests up to a rate of about 200 in. per in. per sec. are presented and discussed for three different steels. The specimens for these tests were taken from plates. The results show that the proportional limit of these materials increases to a value corresponding to the ultimate strength at a strain rate of between 40 and 80 in. per in. per sec. The ultimate strength for all materials tested increases with increasing rate of strain up to a rate of about 100 in. per in. per sec. and then is not affected appreciably.

The influence of rate of strain on the mechanical properties of metals and alloys has been under study by several investigators during the past 10 or 15 years. Most of these investigations have been made at rates that may be obtained with screw-driven machines. The investigations of Winlock and Leiter (**1**)[2] and Elam (**2**) are examples of work which has been done in the field of relatively low strain rates. The investigations of Mann (**3**, **4**), Clark and Datwyler (**5**), Manjoine and Nadai (**6**), Parker and Ferguson (**7**) and Manjoine (**8**) have attempted to reveal the influence of the higher range of strain rate on the mechanical properties of metals. Clark and Duwez (**9**, **10**) have shown that the strain rate in impact tests varies from point to point along the specimen and for a given point it is also dependent upon time. Therefore, the usual tension impact test cannot be employed to study the influence of rate of strain on the properties of metals.

The inability of impact tests to reveal the influence of strain rate may be illustrated by considering a specimen of annealed copper 8 in. long struck at one end, at a velocity of 100 ft. per sec. The rate of strain at the struck end, the middle, and the fixed end of the specimen, as a function of time, can be computed from the strain-time curves. These curves are shown in Fig. 1. In this case the rate of strain reaches a value of 870 in. per in. per sec., while the average rate of strain is 150 in. per in. per sec. With lower impact velocities the maximum strain rate approaches the average strain rate. It is apparent that if one is to study the influence of strain rate on the properties of a material, the test must be

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[2] **The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 575.**

conducted in such a way that the phenomenon of the propagation of plastic strain is not present.

The purpose of this paper is to describe equipment that has been developed to study the influence of pure strain rate on the properties of materials and to present the results of some experiments made with this equipment on three types of steel.

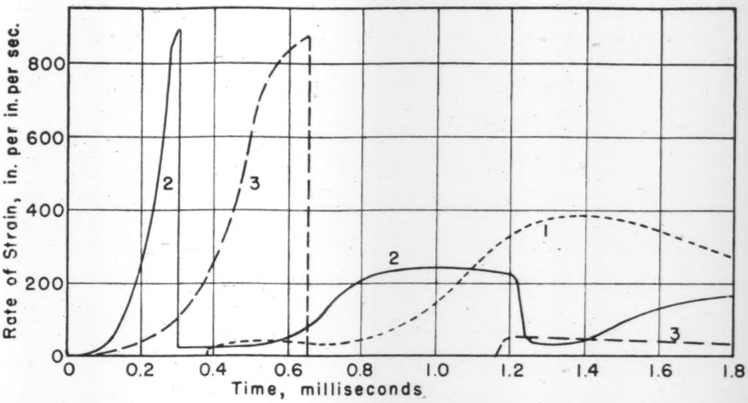


FIG. 1.—Rate of Strain *versus* Time for an Annealed Copper Specimen 8 in. Long Subjected to an Impact Velocity of 100 ft. per sec. (1-Struck End, 2-Middle, 3-Fixed End of Specimen).

EQUIPMENT

Fundamental Considerations:

One method of securing a uniform high strain rate is to use a thin-wall hollow cylindrical specimen in which only circumferential strain is induced by an internal fluid pressure. The ability of such a system to produce a desired constant strain rate depends upon the incompressibility of the fluid employed, the ability to compress the fluid by means of a piston moving at constant velocity, and the stress-strain relation for the material being tested.

It can be shown that the ratio between the actual rate of strain and the ideal rate of strain is given by the following relation:

$$\frac{\frac{d\epsilon}{dt}}{\left(\frac{d\epsilon}{dt}\right)^*} = \frac{1}{1 + \frac{k}{2}\frac{V_f}{V_c}\frac{e}{r}\frac{d\sigma}{d\epsilon}} \quad \ldots (1)$$

with $$\left(\frac{d\epsilon}{dt}\right)^* = \left(\frac{r_p}{r}\right)^2 \frac{1}{2L} v_p \quad \ldots\ldots (2)$$

where:

$\frac{d\epsilon}{dt}$ = rate of strain with compressible fluid,

$\left(\frac{d\epsilon}{dt}\right)^*$ = rate of strain with incompressible fluid,

k = coefficient of compressibility of fluid,

V_f = volume of all fluid within the cylinder,

V_c = volume of the cylinder within the gage section,

e = wall thickness of the specimen,

r = inside radius of the tubular specimen,

r_p = radius of the piston,

$\frac{d\sigma}{d\epsilon}$ = ratio of stress to strain in the specimen,

v_p = velocity of the piston, and

L = length of gage section.

The smaller the value of $\frac{k}{2}\frac{V_f}{V_c}\frac{e}{r}\frac{d\sigma}{d\epsilon}$ in Eq. 1 the closer the rate of strain will be to the ideal rate of strain. The fluid of those most readily available having the lowest compressibility coefficient, $k = 4 \times 10^{-6}$ atmospheres^{-1}, is mercury. The smallest practical value of the fluid volume, V_f, seems to be one and one-half times the volume in the gage length of the specimen, V_c. If thin-wall cylinder theory is to be applied in this case, the maximum value of $\frac{e}{r}$ is about one-tenth.

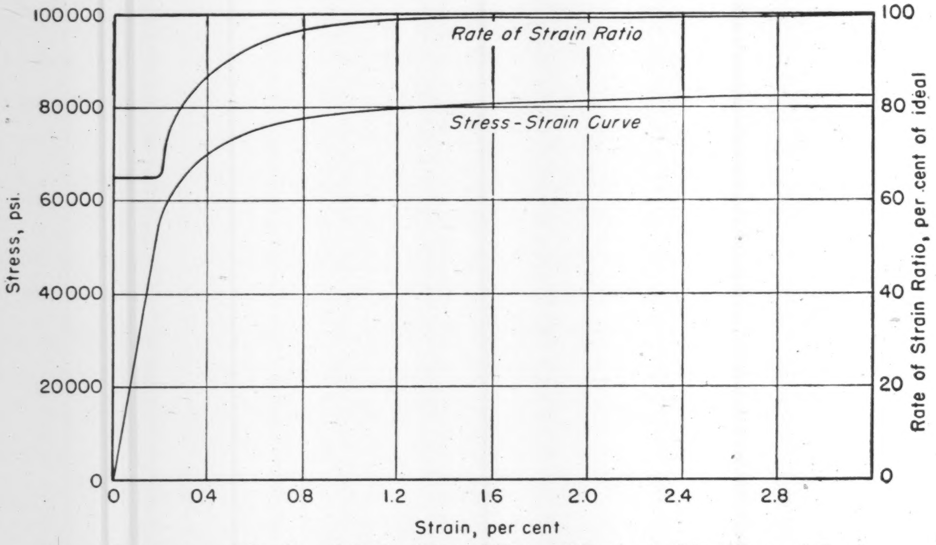


FIG. 2.—Stress *versus* Strain and Ratio of Actual Rate of Strain to Ideal Rate of Strain *versus* Strain for S.A.E. 1020 Cold-Rolled Steel.

The ratio of the actual rate of strain to the ideal rate of strain for a cold-rolled steel can be computed as a function of strain by taking values of k, $\frac{V_f}{V_c}$ and $\frac{e}{r}$ as 4×10^{-6} atmospheres^{-1}, 1.5, and 0.10, respectively, and taking $\frac{d\sigma}{d\epsilon}$ from the stress-strain diagram. The curve resulting from this computation is presented in Fig. 2. This curve shows that in the plastic range the rate of strain will be greater than 90 per cent of the ideal rate.

The range of strain rates obtainable with this method depends upon the dimensions of the specimen and the piston velocity. For a specimen with an inside diameter of 0.250 in., a piston of the same diameter, and a gage length of 1.25 in., the rate of strain is computed by means of Eq. 2 to be 0.48 in. per in. per sec. for a piston velocity of 1 ft. per sec. With a piston velocity of 200 ft. per sec. a rate of strain of about 960 in. per in per sec. can be obtained. Care must be taken with respect to the propagation of elastic waves in the fluid. The maximum piston velocity above which propagation becomes troublesome may be arbitrarily defined as that velocity for which the time required for a pressure wave to travel the length of fluid contained within the specimen is of the same order of magnitude as the time required for the specimen to rupture.

The velocity of propagation of an elastic wave in mercury is of the order of 5000 ft. per sec. With a length of fluid column of approximately 2 in., the time for a wave to travel this distance is 0.03 milliseconds. For a piston velocity of 200 ft. per sec. and a maximum strain of 5 per cent, a specimen of the dimensions previously given will rupture in 0.05 milliseconds. For a piston velocity of 100 ft. per sec., rupture will occur in 0.1 milliseconds and the elastic wave will propagate three times along the fluid. From this information, and from the dimensions employed, it would appear that the propagation in the fluid will not be troublesome if the piston velocity is less than 100 ft. per sec., corresponding to a rate of strain of less than 480 in. per in. per sec. On the basis of these considerations, equipment was designed and constructed, utilizing a tubular type of specimen filled with mercury which is pressurized by means of a piston driven at a desired velocity by impact

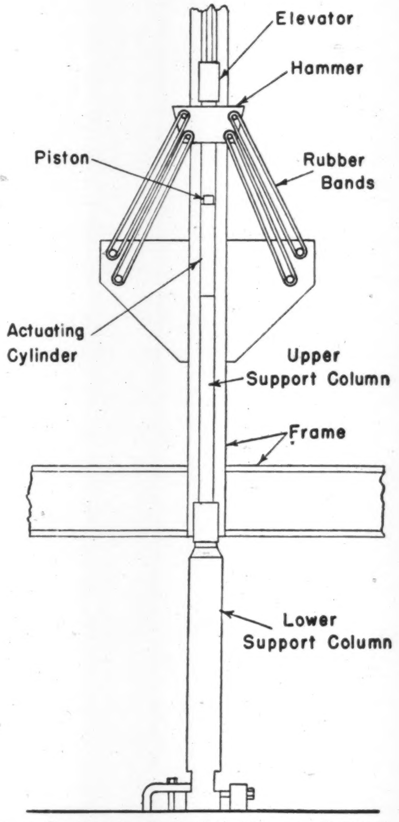

Elevator
Hammer
Piston
Rubber Bands
Actuating Cylinder
Upper Support Column
Frame
Lower Support Column

Fig. 3.—Strain Rate Machine—Side View.

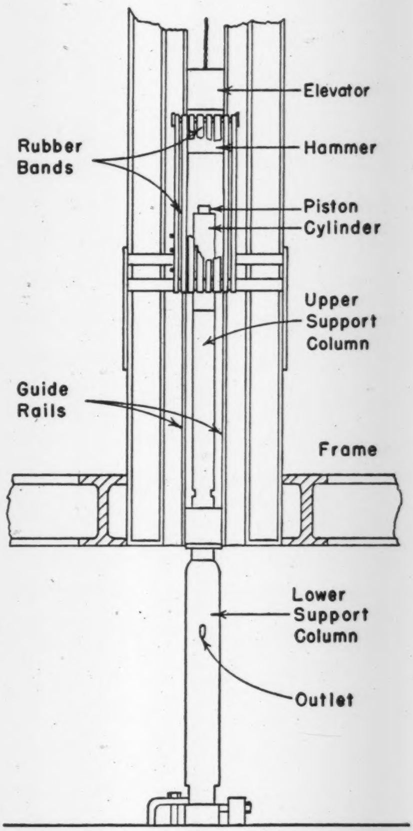

Elevator
Rubber Bands
Hammer
Piston
Cylinder
Upper Support Column
Guide Rails
Frame
Lower Support Column
Outlet

Fig. 4.—Strain Rate Machine—Front View.

Strain Rate Machine:

The frame of the machine used in these investigations consists of two parallel rails 6 in. apart providing guides for a hammer. This frame is supported on large I beams over a pit. In this machine an impacting hammer slides between the two vertical rails and is accelerated by 40 rubber bands $\frac{3}{8}$ by 1 in. The hammer is lifted to the desired height by an electric motor driving through a worm gear reduction, a tandem chain-drive speed reduction, and tandem lifting chains. The lifting chains are attached to a hoisting block which slides between the vertical rails of the machine and grips the top of the impact hammer. A solenoid-operated trigger releases the hammer. The hammer velocity is determined by measuring the time required for the hammer to travel between fixed points of known spacing placed on the frame of the machine near the point of impact. A record of the time intervals is made by means of a cathode-ray oscillograph. Two views of this machine are shown in Figs. 3 and 4.

The assembly holding the specimen and actuating cylinder is threaded into the top end of a column rising from the floor of the pit through a support at the bottom of the rails to a position between the rails as shown in Fig. 4. The lower end of this column rests on a spherical seat and is centered by adjusting screws and clamped into position by bolts.

The actuating cylinder is shown in Fig. 5. The cylinder and piston are made of heat-treated type 440C stainless steel. A free-fitting plug closes the lower end of the specimen and rests on a dynamometer. This assembly is screwed into the column and the inner cylinder and specimen are filled with mercury. The plug transmits the fluid pressure to the dynamometer and eliminates axial restraint in the specimen, thus providing circumferential loading. The dynamometer consists of a short column of heat-treated alloy steel, shown in Fig. 5, to which a resistance-sensitive element is cemented. The elastic deformation of the dynamometer column produced by the fluid force on the plug changes the resistance of the element which causes a change of voltage in the strain-gage circuit. This voltage change is amplified and produces a vertical deflection on a cathode-ray oscillograph. Considering the response of the electrical system and variation of cross-sectional area of the specimen, the stress values obtained are accurate within ± 10 per cent.

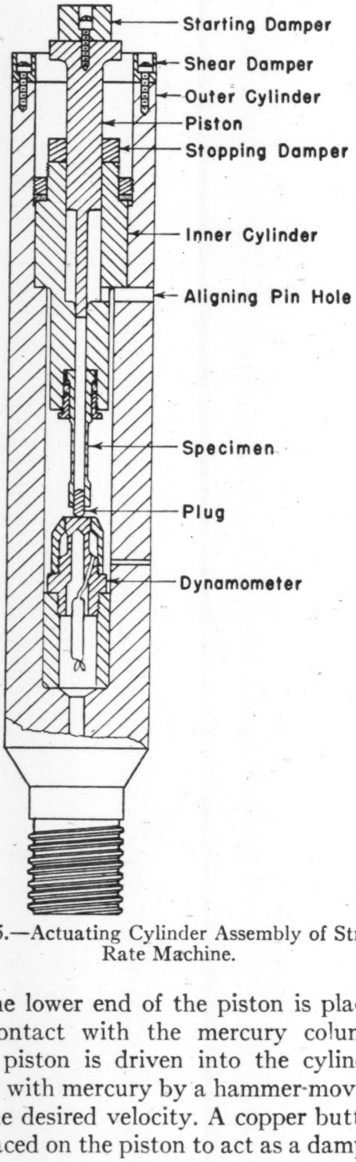


FIG. 5.—Actuating Cylinder Assembly of Strain Rate Machine.

The lower end of the piston is placed in contact with the mercury column. The piston is driven into the cylinder filled with mercury by a hammer moving at the desired velocity. A copper button is placed on the piston to act as a damper to decrease the shock wave.

Two types of hammers are employed.

Strain rates of less than 100 in. per in. per sec. (hammer velocity less than 20 ft. per sec.) are obtained by raising a hammer shown in Fig. 6 to the desired height by means of the elevator and allowing it to fall freely. To attain greater strain rates, the hammer shown in Fig. 7 is used. This hammer is accelerated by the rubber bands previously mentioned. Each hammer is provided with a shear disk of phosphor bronze. When the piston has been driven to the end of its stroke, the impact head of the hammer strikes the rim of the supporting cylinder and the shear disk fails, allowing the hammer to pass downward over the column. The hammer is decelerated by the rubber bands and by a braking system. The drop hammer and rubber band actuated hammer weigh 166 and 48 lb., respectively. Each hammer is designed to have sufficient energy so that the decrease in velocity during the period of applying pressure to the specimen is less than 5 per cent.

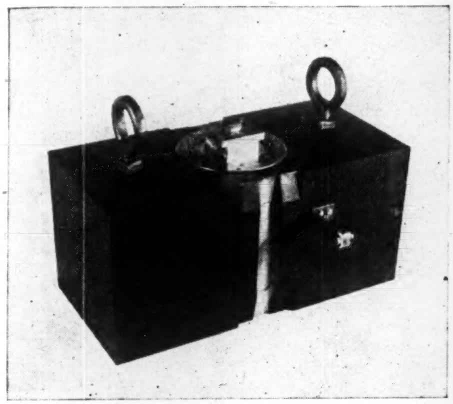


FIG. 6.—Drop Hammer for Strain Rates Less Than 100 in. per in. per sec.

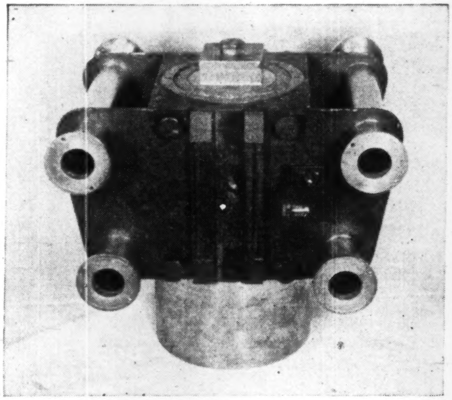


FIG. 7.—Hammer for Strain Rates Greater Than 100 in. per in. per sec.

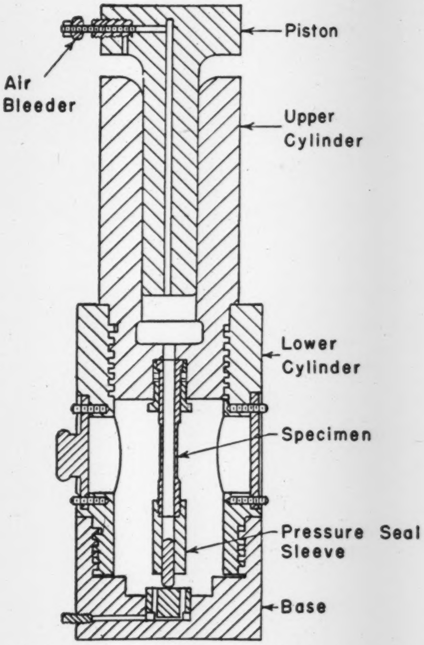
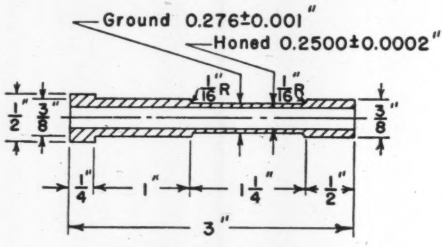


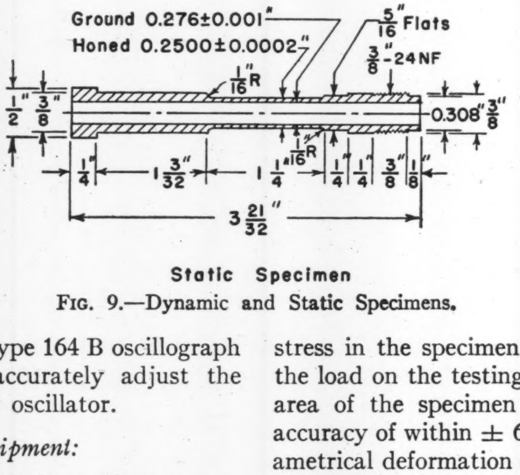
FIG. 8.—Equipment for Static Uniaxial Tests.

While the theoretical limit of pure strain rate with this equipment is 500 in. per in. per sec., the actual limit is about 250 in. per in. per sec. due to the fact that above a velocity of 50 ft. per sec. parts of the cylinder assembly fail under impact loading.

All recording equipment and actuating controls are located at a central panel. This panel contains a standard frequency oscillator, a variable frequency oscillator for each of the two oscillographs, two sweep units, two preamplifiers, a sequence control, calibrating units, a 5-in. Dumont Type 241 oscillograph, 9-in. R.C.A. TMV-168 oscillograph, and power supplies. A camera with 35-mm. film is provided for each oscillograph. A 3-in. Dumont Type 164 B oscillograph is employed to accurately adjust the variable-frequency oscillator.



Dynamic Specimen



Static Specimen

FIG. 9.—Dynamic and Static Specimens.

Static Testing Equipment:

A section view of the equipment employed for the static uniaxial tests is shown in Fig. 8. The specimen is similar to the dynamic specimen except that it is provided with threads at one end onto which a pressure seal is screwed. A closely fitting plug permits free axial motion of the specimen. The operating cylinder with the specimen in place is screwed into the lower cylinder with the free-fitting plug resting on the base of the lower cylinder. The operating cylinder is filled with S.A.E. 70 oil and the piston inserted. The air is bled off through a needle valve in the piston. The equipment is placed in a universal testing machine and pressure applied. Simultaneous values of load and diametrical deformation are recorded. The stress in the specimen is obtained from the load on the testing machine and the area of the specimen with a resulting accuracy of within ± 6 per cent. The diametrical deformation of the specimen is determined by means of a simple linkage mechanism and a Starrett dial gage reading to 0.0001 in. The strain measurements are accurate to within ± 1 per cent.

The usual tension tests were made with a 30,000-lb. universal testing machine with a least reading of 10 lb.

Specimens:

The specimens for static and dynamic uniaxial tests are shown in Fig. 9. The material for the specimens was machined to a diameter of 0.515 in. and a length of 4½ in. A hole was drilled through the blanks along the axis, then reamed and honed to a diameter of 0.2500 ± 0.0002 in. Some holes were hand lapped producing a finish of 4 to 5 microinch rms. Machine honing was necessary in order to finish specimens in large quantitites. The finish obtained varied from 5 to 25 microinch rms. with some holes as rough as 50 microinch rms. The specimens were mounted on an arbor and the outer surface machined. The gage length was ground to a diameter of 0.276 ± 0.001 in. with a resulting surface finish of 5 to 15 microinch rms. The inner and outer diameters of the gage section were concentric within 0.0005 in., and the variation from the nominal hole diameter was 0.0002 in. The over-all accuracy of the determination of wall thickness is within about 7 per cent.

TABLE I.—CHEMICAL COMPOSITION OF PLATES.

	Carbon Steel	Carbon-Manganese Steel				Nickel-Chromium Steel		
Designation	CS	CMn-1	CMn-2	CMn-3	CMn-4	NiCr-1	NiCr-2	NiCr-3
Plate:								
Weight, lb. per sq. ft.	60	25	25	25	60	25	25	25
Thickness, in.	1½	19/32	23/32	19/32	1½	9/16	9/16	9/16
Analysis:								
Carbon, per cent	0.22	0.12	0.17	0.13	0.14	0.36	0.41	0.34
Manganese, per cent	0.55	1.25	1.24	1.23	1.25	0.22	0.21	0.21
Phosphorus, per cent	0.013	0.032	0.031	0.029	0.043	0.017	0.017	0.014
Sulfur, per cent	0.027	0.029	0.028	0.025	0.042	0.020	0.023	0.022
Silicon, per cent	0.22	0.03	0.04	0.23	0.30	0.09	0.08	0.07
Chromium, per cent	0.03	0.02	0.06	0.08	0.07	1.37	1.37	1.38
Nickel, per cent	Trace	0.04	0.08	0.14	Trace	2.73	2.73	3.10
Copper, per cent	Trace	0.20	0.27	0.27	Trace	....	....	....

TABLE II.—RESULTS OF STATIC TENSION TESTS.

Designation	Rockwell Hardness	Proportional Limit, psi.	Ultimate Strength, psi.	Elongation in 8 in., per cent	Reduction of Area, per cent
CS	70.4B	30 500	60 500	19.5	61
		32 000	61 000	18.7	64
CMn-1	82.1B	54 600	73 500	16.2	74
		54 500	73 400	18.2	64
CMn-2	82.7B	54 500	75 100	16.0	75
		54 100	74 800	17.1	75
CMn-3	83.9B	56 500	75 000	14.5	75
		54 000	72 800	16.2	75
CMn-4	84.3B	57 000	73 100	9.7	71
		42 000	73 600	12.9	72
NiCr-1	23.4C	99 000	114 500	8.1	61
		99 000	116 200	9.5	61
NiCr-2	22.9C	99 000	114 000	8.1	61
		99 000	114 200	8.1	61
NiCr-3	23.6C	97 000	116 000	9.3	69
		97 500	115 200	9.4	69

The static tension test specimens, 0.300 in. in diameter with a gage length of 8.0 in., were used in determining the usual static tensile properties.

MATERIALS TESTED

The materials considered in this investigation consisted of three types, namely, a carbon steel, a low-carbon manganese steel, and a nickel-chromium steel. These materials were in the form of plates varying from $\frac{9}{16}$ to 2 $\frac{3}{8}$ in. in thickness. The analyses of the plates are given in Table I. The specimen blanks were sawed from the plates so that the axes of the specimens were parallel to the direction of rolling. On the thicker plates, the blanks were split parallel to the plane of rolling so that the test section of the specimens would not include material in the central plane of the plate.

EXPERIMENTAL RESULTS

Static Test Results:

Static tension tests and static tests using a tubular specimen under internal pressure were performed on the

TABLE III.—RESULTS OF STATIC UNIAXIAL TESTS.

Designation	Specimen Number	Profilometer Reading, microinch rms.	Outside Diameter, in.	Inside Diameter, in.	Gage Length, in.	Proportional Limit, psi.	Ultimate Strength, psi.	Uniform Strain at Rupture, per cent
CS	2	20	0.2763	0.2500	1.25	45 000	63 200	4.5
	3	19	0.2763	0.2500	1.25	45 000	64 000	6.5
	4	14	0.2762	0.2500	1.25	35 500	59 000	5.4
	5	10	0.2750	0.2498	1.25	38 000	60 000	4.1
	6	8	0.2750	0.2498	1.25	36 000	56 000	2.9
	Average					39 900	60 400	4.6
CMn 1	1	9	0.2760	0.2516	1.25	52 000	78 000	4.3
	2	8	0.2770	0.2505	1.25	52 500	77 200	9.4
	3	8	0.2750	0.2498	1.25	50 000	73 000	4.7
	4	11	0.2747	0.2498	1.25	52 000	70 000	2.6
	Average					51 600	74 450	5.2
CMn-2	1	9	0.2777	0.2522	1.25	55 000	81 500	12.6
	2	10	0.2775	0.2516	1.25	53 500	80 500	12.4
	3	6	0.2746	0.2498	1.25	52 000	74 000	8.0
	4	7	0.2740	0.2498	1.25	54 500	77 000	4.4
	Average					53 700	78 200	9.4
CMn-3	1	..	0.2760	0.2500	1.25	52 500	85 000	11.6
	2	..	0.2760	0.2500	1.25	56 500	82 500	18.2
	3	..	0.2765	0.2500	1.25	51 500	83 000	16.0
	4	..	0.2760	0.2500	1.25	52 000	88 000	8.6
	Average					52 500	84 600	13.6
CMn-4	2	10	0.2813	0.2548	1.25	57 500	80 000	6.0
	3	9	0.2780	0.2520	1.25	55 000	78 000	7.7
	4	..	0.2930	0.2670	1.25	46 000	70 500	3.0
	Average					52 800	76 200	5.6
NiCr-1	1	10	0.2813	0.2550	1.25	84 000	105 000	12.0
	2	10	0.2777	0.2516	1.25	96 000	115 500	6.0
	3	9	0.2815	0.2548	1.25	92 000	116 700	7.5
	4	10	0.2773	0.2516	1.25	95 000	116 500	7.4
	Average					91 800	113 400	8.2
NiCr-2	1	9	0.2810	0.2548	1.25	89 000	118 000	3.5
	2	11	0.2776	0.2516	1.25	94 000	107 500	2.2
	3	8	0.2783	0.2520	1.25	94 000	119 500	5.4
	Average					92 300	115 000	3.7
NiCr-3	1	..	0.2760	0.2500	1.25	110 000	128 000	6.0
	2	..	0.2765	0.2500	1.25	105 500	126 000	4.5
	Average					107 800	127 000	5.2

TABLE IV.—RESULTS OF STRAIN RATE TESTS ON CS PLATE.

Specimen	Profilometer Reading, microinch rms.	Outside Diameter, in.	Inside Diameter, in.	Gage Length, in.	Velocity of Hammer, ft. per sec.		Rate of Strain, in. per in. per sec.	Proportional Limit, psi.	Ultimate Strength, psi.	Outside Diameter after Rupture, in.	Maximum Uniform Strain at Rupture, per cent
					Free Fall, computed	Rubber accelerated, measured					
No. 8	6	0.277	0.250	1.50	10	....	35.4	49 000	61 000	0.286	3.1
No. 4	13	0.275	0.250	1.25	10	....	42.3	54 400	68 500	0.285	3.4
No. 5	15	0.275	0.250	1.25	10	....	42.3	56 200	67 500	0.285	3.6
No. 6	12	0.275	0.250	1.25	10	....	42.3	54 000	70 000	0.293	6.5
No. 7	24	0.275	0.250	1.25	10	....	42.3	53 000	67 000	0.297	8.0
No. 9	6	0.277	0.250	1.50	18	....	63.5	......	67 000	0.293	5.8
No. 20	8	0.274	0.250	1.25	18	....	76.2	......	82 000	0.281	2.6
No. 12	6	0.278	0.250	1.25	..	25.1	106.0	......	76 000	0.286	2.9
No. 15	10	0.277	0.250	1.25	..	42.1	178.0	......	83 000	0.292	5.4
No. 16	11	0.277	0.250	1.25	..	42.8	181.0	......	90 000	0.290	4.7
No. 17	5	0.277	0.250	1.25	..	43.5	184.0	......	89 000	0.292	5.4

TABLE V.—RESULTS OF STRAIN RATE TESTS ON CMn PLATE.

Specimen	Profilometer Reading, microinch rms.	Outside Diameter, in.	Inside Diameter, in.	Gage Length, in.	Velocity of Hammer, ft. per sec.		Rate of Strain, in. per in. per sec.	Proportional Limit, psi.	Ultimate Strength, psi.	Outside Diameter after Rupture, in.	Maximum Uniform Strain at Rupture, per cent
					Free Fall, computed	Rubber accelerated, measured					
					CMn-1						
No. 1	10	0.275	0.250	1.25	10	....	42.3	67 500	80 000	0.287	4.4
No. 2	8	0.276	0.250	1.25	10	....	42.3	67 300	80 000	0.299	8.7
No. 3	9	0.277	0.250	1.25	10	....	52.3	61 600	77 200	0.297	7.2
No. 4	9	0.277	0.250	1.25	10	....	42.3	63 000	75 000	0.294	6.1
No. 5	10	0.278	0.250	1.25	18	....	76.2	......	75 700	0.290	4.3
No. 6	10	0.277	0.250	1.25	18	....	76.2	......	81 600	0.293	5.8
No. 7	9	0.277	0.250	1.25	18	....	76.2	......	82 500	0.283	2.2
No. 8	10	0.278	0.250	1.25	18	....	76.2	......	74 500	0.294	5.8
No. 11	6	0.277	0.250	1.25	..	24.0	101.5	......	108 000	0.283	2.2
No. 9	8	0.277	0.250	1.25	..	25.2	106.8	......	100 000	0.296	6.9
No. 10	10	0.277	0.250	1.25	..	27.3	115.5	......	96 000	0.285	2.9
No. 17	..	0.275	0.250	1.25	..	43.1	182.5	......	98 000	0.288	4.7
No. 18	..	0.275	0.250	1.25	..	43.7	185.0	......	102 000	0.282	2.5
No. 12	5	0.278	0.250	1.25	..	44.3	187.0	......	115 000	0.283	1.8
					CMn-2						
No. 5	4	0.275	0.250	1.50	10	....	35.4	67 000	75 600	0.300	9.1
No. 3	17	0.275	0.250	1.25	10	....	42.3	60 000	76 500	0.289	5.1
No. 4	6	0.277	0.250	1.25	10	....	42.3	56 000	65 700	0.295	6.5
No. 6	11	0.277	0.250	1.25	10	....	42.3	64 000	78 200	0.295	6.5
No. 7	9	0.278	0.250	1.25	10	....	42.3	56 000	66 000	0.288	3.6
No. 18	9	0.274	0.250	1.25	10	....	42.3	......	75 000	0.295	7.7
No. 8	9	0.277	0.250	1.25	18	....	76.2	......	86 500	0.306	10.5
No. 9	10	0.277	0.250	1.25	18	....	76.2	......	81 500	0.302	9.0
No. 10	11	0.278	0.250	1.25	18	....	76.2	......	80 500	0.305	9.7
No. 19	11	0.275	0.250	1.25	18	....	76.2	......	90 000	0.298	8.4
No. 13	..	0.280	0.255	1.25	..	25.1	101.5	......	81 000	0.288	2.9
No. 11	8	0.278	0.250	1.25	..	24.7	104.5	......	76 500	0.285	2.5
No. 14	10	0.274	0.250	1.25	..	25.5	108.0	......	84 000	0.284	3.6
No. 17	10	0.275	0.250	1.25	..	41.6	176.0	......	97 000	0.290	5.5
No. 16	9	0.275	0.250	1.25	..	41.7	176.1	......	106 000	0.292	6.2
No. 15	9	0.275	0.250	1.25	..	46.6	197.0	......	100 000	0.291	5.8
					CMn-3						
No. 9	8	0.281	0.255	1.25	10	....	41.6	66 000	86 000	0.293	4.3
No. 10	7	0.281	0.255	1.25	10	....	41.6	74 000	102 000	0.298	6.0
No. 11	8	0.280	0.255	1.25	10	....	41.6	81 000	97 000	0.292	4.3
No. 12	9	0.281	0.255	1.25	18	....	73.3	......	101 000	0.296	5.3
No. 13	8	0.281	0.255	1.25	18	....	73.3	......	104 000	0.295	5.0
No. 14	3	0.280	0.255	1.25	18	....	73.3	......	101 000	0.296	5.7
No. 1	8	0.280	0.255	1.25	..	26.1	106.0	......	98 500	0.303	8.2
No 2	11	0.281	0.255	1.25	..	26.4	107.0	......	95 000	0.293	4.3
No. 3	5	0.280	0.255	1.25	..	26.6	108.0	......	94 000	0.303	8.2
No. 8	9	0.281	0.255	1.25	..	41.6	169.0	......	100 000	0.294	4.6
No. 7	7	0.281	0.255	1.25	..	42.5	173.0	......	103 000	0.295	5.0
No. 5	5	0.281	0.255	1.25	..	44.0	179.0	......	100 000	0.293	5.3
					CMn-4						
No. 7	8	0.277	0.250	1.25	10	....	42.3	61 500	70 500	0.295	6.5
No. 9	8	0.277	0.250	1.25	10	....	42.3	56 500	68 000	0.295	6.5
No. 6	13	0.278	0.250	1.25	10	....	42.3	56 000	68 500	0.289	4.0
No. 22	8	0.274	0.250	1.25	18	....	76.2	9 500	100 500	0.293	6.9
No. 23	9	0.274	0.250	1.25	18	....	76.2	9 100	96 000	0.291	6.2
No. 21	9	0.275	0.250	1.25	18	....	76.2	86 000	96 000	0.297	8.0
No. 16	11	0.278	0.250	1.25	..	27.2	115.0	......	95 000	0.292	5.0
No. 19	9	0.274	0.250	1.25	..	40.1	170.0	......	95 000	0.282	2.9
No. 20	9	0.275	0.250	1.25	..	42.9	181.5	......	104 000	0.302	9.8

three different types of materials. The results of the static tension tests are presented in Table II. The results of the static tests made on tubular specimens are presented in Table III. The results presented in Table III were obtained from the curves of hoop stress *versus* hoop strain. In making the static tests on the tubular specimens a few specimens developed a fine longitudinal crack shortly after reaching the proportional limit causing the oil to leak and the inside pressure to drop to zero. All specimens from some plates, not reported in this paper, developed longitudinal cracks in the static tests.

TABLE VI.—RESULTS OF STRAIN RATE TESTS OF NiCr PLATE.

Specimen	Profilometer Reading, microinch rms.	Outside Diameter, in.	Inside Diameter, in.	Gage Length, in.	Velocity of Hammer, ft. per sec.		Rate of Strain, in. per in. per sec.	Proportional Limit, psi.	Ultimate Strength, psi.	Outside Diameter after Rupture, in.	Maximum Uniform Strain at Rupture, per cent
					Free Fall, computed	Rubber accelerated, measured					
					NiCr-1						
No. 6.....	12	0.277	0.250	1.50	10	....	35.4	74 000	116 000	0.298	7.6
No. 3.....	10	0.274	0.250	1.25	10	....	42.3	80 000	111 000	0.283	3.3
No. 7.....	7	0.277	0.250	1.25	10	....	42.3	79 500	108 000	0.300	8.3
No. 8.....	10	0.277	0.250	1.25	18	....	76.2	......	104 000	0.296	6.9
No. 9.....	10	0.277	0.250	1.25	18	....	76.2	......	109 000	0.298	7.6
No. 10.....	10	0.277	0.250	1.25	18	....	76.2	......	118 000	0.298	7.6
No. 18.....	10	0.290	0.264	1.25	..	27.3	104.0	......	145 000	0.296	2.1
No. 12.....	7	0.277	0.250	1.25	..	24.7	104.5	......	119 000	0.284	2.5
No. 11.....	8	0.277	0.250	1.25	..	25.6	108.2	......	130 000	0.287	3.6
No. 13.....	13	0.277	0.250	1.25	..	26.6	112.5	......	149 000	0.302	9.0
No. 15.....	9	0.277	0.250	1.25	..	44.0	186.5	......	148 000	0.292	5.4
No. 14.....	12	0.277	0.250	1.25	..	44.7	189.0	......	163 000	0.298	7.6
					NiCr-2						
No. 4.....	5	0.277	0.250	1.50	10	....	35.4	74 000	101 500	0.292	5.4
No. 5.....	8	0.278	0.250	1.50	10	....	35.4	79 600	107 000	0.295	6.1
No. 2.....	15	0.276	0.250	1.25	10	....	42.3	85 200	108 500	0.282	2.2
No. 3.....	13	0.276	0.250	1.25	10	....	42.3	88 200	105 000	0.291	5.4
No. 6.....	5	0.278	0.250	1.50	18	....	63.5	......	112 000	0.300	7.9
No. 7.....	6	0.277	0.250	1.50	18	....	63.5	......	117 000	0.292	5.4
No. 8.....	9	0.277	0.250	1.25	18	....	76.2	......	119 000	0.290	4.7
No. 9.....	13	0.278	0.250	1.25	18	....	76.2	......	132 000	0.293	5.4
No. 16.....	..	0.301	0.275	1.25	..	25.4	89.0	......	144 000	0.318	5.6
No. 17.....	..	0.287	0.260	1.25	..	25.6	108.5	......	130 000	0.293	2.1
No. 10.....	11	0.277	0.250	1.25	..	27.0	114.0	......	147 000	0.300	8.3
No. 14.....	13	0.277	0.250	1.25	..	44.7	189.0	......	146 000	0.290	4.7
No. 15.....	7	0.277	0.250	1.25	..	46.1	195.0	......	153 000	0.291	5.1
No. 13.....	9	0.278	0.250	1.25	..	48.2	204.0	......	156 000	0.290	4.3
					NiCr-3						
No. 7.....	8	0.281	0.255	1.25	10	....	41.6	120 000	150 000	0.297	5.7
No. 8.....	5	0.280	0.255	1.25	10	....	41.6	115 000	150 000	0.292	4.3
No. 9.....	8	0.280	0.255	1.25	10	....	41.6	112 000	146 000	0.292	4.3
No. 10.....	7	0.280	0.255	1.25	18	....	73.3	......	150 000	0.293	4.6
No. 11.....	6	0.280	0.255	1.25	18	....	73.3	......	134 000	0.295	5.4
No. 2.....	7	0.281	0.255	1.25	..	24.3	99.0	......	131 000	0.293	4.3
No. 3.....	7	0.281	0.255	1.25	..	26.6	104.5	......	126 000	0.292	3.9
No. 1.....	6	0.280	0.255	1.25	..	26.2	106.5	......	132 000	0.298	6.4
No. 5.....	5	0.279	0.255	1.25	..	43.1	175.5	......	136 000	0.293	5.0
No. 4.....	8	0.280	0.255	1.25	..	44.0	179.0	......	123 000	0.296	5.7
No. 6.....	8	0.279	0.255	1.25	..	47.5	193.0	......	135 000	0.295	5.7

The appearance of fine cracks associated with premature rupture of the specimens may be due to the presence of impurities which give the steel a fiber structure in the longitudinal direction. The importance of this factor is increased in the case of specimens having very thin walls (0.01 in.) such as used in this investigation.

It may be noted that for specimens of

the carbon steel presented in Table III the proportional limit varies between 35,500 and 45,000 psi. and the ultimate strength varies between 56,000 and 64,000 psi. This rather large scatter cannot be accounted for by the inaccuracy of the load measurements. It is probable that the lack of structural uniformity between specimens having such thin walls is the principal reason for the scattered results. The results obtained from the other two materials are more

TABLE VII.—RESULTS OF IZOD IMPACT TESTS.

Material	Notch[a]	Energy Absorbed, ft-lb.					Average Energy Absorbed, ft-lb.	Grand Average Energy Absorbed, ft-lb.
		Specimen No. 1	Specimen No. 2	Specimen No. 3	Specimen No. 4	Specimen No. 5		
CS	No. 1	57.5	77.5	77.5	77.5	46.3	67.5	
	No. 2	46.5	51.0	50.0	45.5	47.4	48.1	
	No. 3	79.0	72.5	76.0	71.5	65.6	72.9	62.8
CMn-1	No. 1	92.8	93.5	96.0	94.0	93.0	93.9	
	No. 2	91.0	91.5	89.0	91.0	94.0	91.3	
	No. 3	98.0	100.0	95.5	98.0	102.0	98.7	93.4
CMn-2	No. 1	96.0	96.0	94.0	94.5	102.0	96.5	
	No. 2	96.5	61.5	100.0	61.0	91.0	82.0	
	No. 3	97.0	96.5	102.0	98.0	77.5	94.2	91.9
CMn-3	No. 1	96.0	98.5	98.0	95.0	98.0	97.1	
	No. 2	92.3	90.0	96.0	92.0	102.0	94.5	
	No. 3	105.0	102.0	105.0	106.0	105.0	104.6	98.6
CMn-4	No. 1	22.5	19.0	38.0	43.5	51.8	35.0	
	No. 2	23.0	42.0	37.0	39.0	42.4	36.7	
	No. 3	32.0	37.0	50.5	32.5	60.2	42.4	38.0
NiCr-1	No. 1	67.0	63.0	63.0			64.3	
	No. 2	65.5	66.5	72.0			68.0	
	No. 3	62.7	65.5	63.0			63.7	65.3
NiCr-2	No. 1	64.0	68.0	61.5			63.0	
	No. 2	64.5	66.5	65.5			65.5	
	No. 3	59.0	60.0	62.5			60.5	63.5
NiCr-3	No. 1	81.0	80.0	80.5			80.5	
	No. 2	85.5	89.0	81.0			85.8	
	No. 3	83.0	81.0	81.0			81.6	82.6

[a] Notches Nos. 1 and 3 are parallel to the plane of the plate, while Notch No. 2 is perpendicular to the plate.

TABLE VIII.—COMPARISON BETWEEN STATIC AND DYNAMIC VALUES OBTAINED WITH TUBULAR SPECIMENS AND WITH TENSION SPECIMENS.

Designation of Plate	Results of Tests on Tubular Specimens				Results of Tensile Tests			
	Static Proportional Limit, psi.	Static Ultimate Strength, psi.	Ultimate Strength for Rate of Strain of about 190 in. per in. per sec., psi.	Increase in Ultimate Strength, per cent	Static Proportional Limit, psi.	Static Ultimate Strength, psi.	Dynamic Ultimate Strength, psi.	Increase in Ultimate Strength, per cent
CS	39 900	60 400	87 000	45	......	......	......	..
CMn-1	51 600	74 550	105 000	41	54 550	73 450	99 500	35
CMn-2	53 750	78 250	101 000	29	54 300	74 950	102 000	36
CMn-3	52 500	84 625	104 000	23	55 250	73 900	100 000	35
CMn-4	52 800	76 200	99 500	31	......	......	......	..
NiCr-1	91 750	113 425	155 500	37	99 250	115 350	142 000	23
NiCr-2	92 333	115 000	151 000	31	99 000	114 100	144 000	26
NiCr-3	107 750	127 000	131 300	3	97 250	115 650	146 500	27

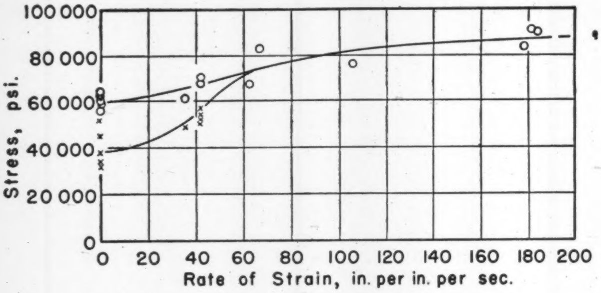


FIG. 10.—Ultimate Strength (o) and Proportional Limit (×) *versus* Rate of Strain for Carbon Steel Plate (CS).

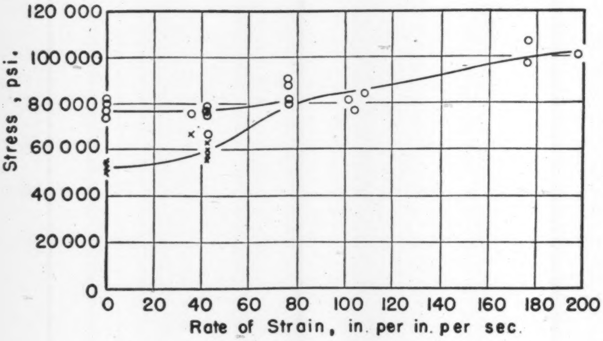


FIG. 11.—Ultimate Strength (o) and Proportional Limit (×) *versus* Rate of Strain for Carbon Manganese Steel Plate (CMn-2).

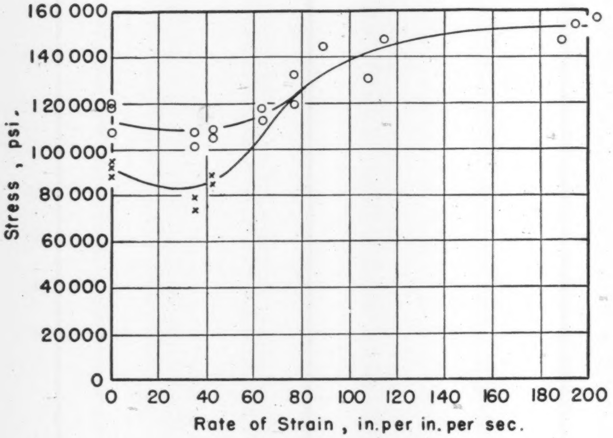


FIG. 12.—Ultimate Strength (o) and Proportional Limit (×) *versus* Rate of Strain for Nickel Chromium Steel Plate (NiCr-2).

consistent and the number of specimens in which a fine crack developed during the test was relatively small. The microstructure of these two materials is much more uniform and seems to present less directional inhomogeneity than that of the carbon steel.

Strain Rate Test Results:

The results of dynamic tests made on the three materials are presented in Tables IV, V, and VI. The rate of strain in the specimen has been computed according to Eq. 2 and is expressed in in. per in. per sec. The values of ultimate strength and proportional limit have been computed from the force-time diagrams.

The maximum fluid pressure is computed from the value of the maximum force recorded in the test. From this the ultimate strength is computed by means of the formula $\sigma = p \dfrac{r_a}{e}$ where p is the pressure, e the wall thickness, and r_a the average radius of the cylinder. When the recorded diagram presents some oscillations, a smooth curve is traced through an average value and the maximum force is read from this smooth curve. The proportional limit is taken as the stress corresponding to the force at which the recorded diagram starts to curve.

The diameter of the specimen was measured after the test. This measurement was made on that part of the specimen which remained nearly cylindrical in shape and not in the region of the fracture. In general, that portion of the specimen which remained almost cylindrical after rupture and was used for the measurement of the uniform maximum hoop strain at rupture occupied more than one-half the gage length.

The ultimate strength and the proportional limit are plotted against the rate of strain for typical cases of each material in Figs. 10 to 12 inclusive.

In the case of the carbon steel, the ultimate strength appears to increase gradually with the rate of strain from an average static value of 60,400 psi. to approximately 87,300 psi. for a rate of strain of about 180 in. per in. per sec. The proportional limit for rates of strain of about 40 in. per in. per sec. is slightly higher than the average static value. For higher rates of strain the force-time diagram and consequently the stress-strain diagram is almost a horizontal straight line in the plastic range, hence the proportional limit is taken equal to the ultimate strength. The maximum uniform strain of this material at rupture, as indicated in Table IV, varies from 2.6 to 8.0 per cent and does not seem to show any systematic variation with rate of strain. It is important to consider that the very small wall thickness of the specimens makes it difficult to reproduce rupture conditions from specimen to specimen.

In the case of the manganese steel, the ultimate strength appears to increase with increasing rate of strain from about 78,000 psi. under static conditions to about 100,000 psi. for a rate of strain of approximately 200 in. per in. per sec. The proportional limit appears to become equal to the ultimate strength for rates of strain above approximately 70 in. per in. per sec. In the case of this material, the uniform strain at rupture, as indicated in Table V, varies from 1.8 to 10.5 per cent. As in the case of the previous material discussed there is no apparent relation between these values and the rate of strain.

For specimens of the nickel-chromium steel, the ultimate strength appears to increase more or less with the rate of strain, but the results are more scattered than those obtained for the other two materials. For one plate of this material (NiCr-2) there is a slight decrease of ultimate strength at a rate of strain of about 40 in. per in. per sec. The general increase in ultimate strength is from about 115,000 psi. under static conditions to about 145,000 psi. for a rate of strain of approximately 200 in. per in. per sec. At a rate of strain of approximately 60 in. per in. per sec., the proportional limit appears to reach the ultimate strength. The maximum uniform strain at rupture is scattered between 2.1 and 9.0 per cent and does not seem to be influenced by rate of strain.

Notched-Bar Impact Tests:

In addition to the strain rate tests, notched-bar impact tests were made on specimens of each plate. These results are presented in Table VII.

DISCUSSION OF RESULTS

For most plates from which samples were taken, the ultimate strength increases with increasing strain rate. In some instances the increase is not very great. For purposes of comparison the average results of the static tests and dynamic tests, the latter at a strain rate of about 190 in. per in. per sec., are summarized in Table VIII. In this table the increase of ultimate strength due to the rate of strain is expressed in per cent of the static value. These results show that the increase of ultimate strength is of the order of 45 per cent for the carbon steel, about 30 per cent for the manganese steel, and about 30 per cent for the nickel-chromium steel with the exception of the group of samples from one plate (NiCr-3) in which case there is practically no change of ultimate strength.

The proportional limit of all these materials increases with rate of strain. For rates of strain greater than about 70 in. per in. per sec., the recorded stress-strain diagrams were practically horizontal in the plastic range, and the values taken for proportional limit are then equal to the ultimate strength.

The maximum uniform strain at rupture is quite scattered as observed in both the static and dynamic tests. For this reason no systematic relation can be found between the uniform strain at rupture and the rate of strain. It is probable, as pointed out previously, that the conditions which initiate rupture are not easily reproduced because of the importance of structural inhomogeneity of the steel and the small wall thickness.

The average values of the static proportional limit and static ultimate strength determined with tubular specimens are about the same as those determined in the static tension tests.

The ultimate tensile strength of some of these materials was determined by means of impact tests in the manner reported in a previous paper (11). As can be seen in Table VIII, the values of the average ultimate strength corresponding to a rate of strain of about 190 in. per in. per sec. as determined by the tests presented in this paper and the average ultimate tensile strength determined under impact conditions are of the same order of magnitude. In comparing the results of tensile impact tests with those obtained from the strain rate tests, it must be remembered that the comparison cannot be established in terms of rate of strain. It has been shown that in tensile impact tests the actual rate of strain varies with both the position of the point along the specimen and the time. The rate of strain is theoretically infinite at the point reached by the front of the stress wave and has values actually much higher than the average rate of strain computed as if no wave propagation existed. This explains why the ultimate strength determined from tensile impact tests with impact velocities ranging from 25 to 200 ft. per sec. are apparently independent of the impact velocity.

The close correspondence of ultimate strength determined by means of the strain rate tests and the impact tests may be interpreted on the basis that the effect of an impact on a tension specimen is about equivalent to a rate of strain of the order of 200 in. per in. per sec., in spite of the fact that the actual rate of strain produced by the impact may reach, at some point, and at certain times, much higher values. This gives some support to the belief that at strain rates appreciably above 200 in. per in. per sec. the ultimate strength is not altered markedly. Therefore, if strain rates greater than 200 in. per in. per sec. are encountered in a structural member, the results of tests made at a rate of about 200 in. per in. per sec. will give a reliable evaluation of proportional limit and ultimate strength. However, if higher strain rates affect the elongation adversely this test will not give the desired results.

Acknowledgment:

The investigation presented in this paper was conducted at California Institute of Technology under a contract with the National Defense Research Committee of the Office of Scientific Research and Development. The authors wish to acknowledge the assistance of D. S. Wood, who was largely responsible for the design of the equipment used in these tests, and H. E. Martens and D. A. Elmer, who conducted most of the tests presented in this paper.

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DISCUSSION

Mr. T. J. Dolan.[1]—I should like to ask a question about the values of proportional limit reported in the paper. It is my impression that the photographic record of the oscilloscope trace consists of a pressure-time diagram. Do I understand that the proportional limits plotted in the paper are the values scaled from those diagrams (rather than from the commonly used stress-strain diagram)? Were the strain rates listed on the figures the actual measured values or were these computed strain rates based on the velocity of movement of the mercury column?

It seems possible that some questions might arise as to whether pressure pulses in the column of mercury would alter that average strain rate during the time of the test at localized spots and perhaps lead to unpredictable conditions.

Mr. D. S. Clark (*author*).—The proportional limit is taken from the force-time diagram obtained from the dynamometer at the end of the specimen. The pressure pulses that you are talking about are the limiting factors in the design of this equipment.

The strain rates listed on the figures are based on the velocity of the plunger acting on the mercury.

It is mentioned in the paper that the limit of the machine is of the order of 500 in. per in. per sec. That is more or less the theoretical limit as computed from the propagation of pulses in the mercury column.

If we were to exceed that rate of strain, we then would get into trouble with localized pressure regions, which would nullify our results. We have actually kept well below that limit. The details of the design from that point of view are considered in detail in the paper.

Mr. H. F. Moore.[2]—In determining the proportional limit, how did the authors determine the point where the stress-strain diagram ceased to be a straight line and became a curve? Or was the proportional limit determined by the intersection of some "offset" with the stress-strain curve?

Mr. Clark.—Possibly we were more arbitrary in selecting the proportional limit than we should have been, but we took the stress at which the slope of the force-time curve changed. We may have been incorrect in calling it a true proportional limit, but it is practical to call it a proportional limit. Possibly we should define it clearly as the breaking away from the linear portion of the curve.

Mr. Moore.—Have you any evidence that there is a true proportional limit?

Mr. Clark.—No. I have more evidence, I think, that there is not.

Mr. R. L. Templin[3].—I should like to ask the authors of this very interesting paper whether they tested a few specimens to check the anisotropy of these plates.

Mr. Clark.—These specimens were all taken in the same direction in the plate. We should like to have made tests in other directions as well, but with the time required for testing and the cost of specimens, this was not feasible.

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[3] Ass't. Director of Research and Chief Engineer of Tests, Research Laboratories, Aluminum Company of America, New Kensington, Pa.

THE INFLUENCE OF SPECIMEN DIMENSION AND SHAPE ON THE RESULTS IN TENSION IMPACT TESTING*

BY D. S. CLARK[1] AND D. S. WOOD[1]

SYNOPSIS

A number of papers have been presented to reveal the influence of impact velocity on the tensile properties of materials. Most investigators have not reported the existence of a critical impact velocity. This paper presents data to show that it is necessary to maintain a ratio of specimen length to diameter of at least 13 in order clearly to reveal a critical velocity. The results of tests are reported to show that for a constant cross-sectional area the shape of the cross-section of the specimen does not alter the tensile impact characteristics of a material.

Within the past ten or fifteen years, several investigators (**1** to **7**)[2] have reported the results of tension impact tests in which test specimens of differing dimensions were employed. In those studies the propagation of elastic and plastic strain waves was not taken into account in interpreting the data. The results of tension impact tests in the light of strain propagation have been discussed by Duwez and Clark (**8**,**9**). This latter work served as a basis for the study of the tensile impact properties of several metals and alloys by Clark and Wood (**10**). In this last paper the existence of a true critical impact velocity was demonstrated in several metals and justified on the basis of the theory of plastic strain propagation (**11**). Prior to this, true critical velocities had not been reported. This is of interest since others have made tests of this character; why did they not observe this phenomenon?

The observance of a critical impact velocity may depend upon the length of the specimen used in the test and also upon the shape and size of the cross-section. It is the purpose of this paper to present data of importance to this question.

EQUIPMENT

The equipment used in the performance of the tests reported in this paper has been described in previous papers (**4**,**8**,**9**,**10**). Briefly, the equipment consists of a rotating wheel provided with impacting jaws which make contact with one end of a specimen after the wheel has attained the desired velocity. The machine is capable of a peripheral velocity of slightly greater than 200 ft. per sec. The forces acting on the fixed end of the specimen are determined by means of a dynamometer in conjunction with a cathode ray oscilloscope which provides a record of force *versus* time.

The static tension tests reported in this paper were made with a 30,000-lb. universal testing machine having a least reading of 10 lb.

THE INFLUENCE OF RATIO OF LENGTH TO DIAMETER

The specimens for the investigation of the influence of the ratio of length to

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Associate Professor of Mechanical Engineering and Lecturer, respectively, California Institute of Technology, Pasadena, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 585.

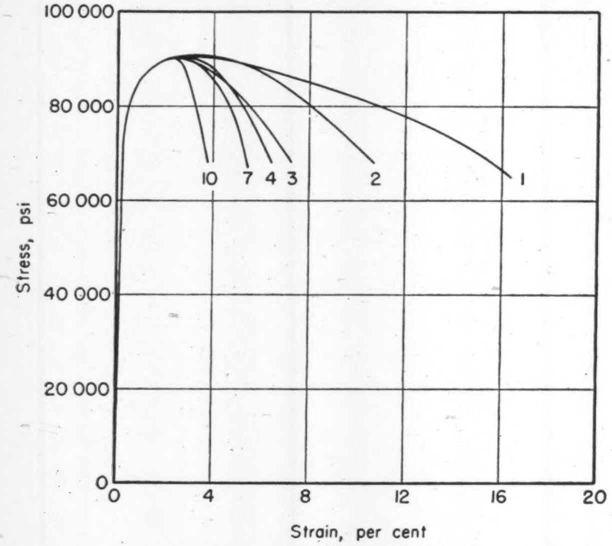

FIG. 1.—Static Stress-Strain Curves for S.A.E. 1020 Cold-Rolled Steel (all specimens are 0.300 in. in diameter).

TABLE I.—RESULTS OF STATIC TESTS ON SPECIMENS OF S.A.E. 1020 COLD-ROLLED STEEL.

Gage Length, in.	Proportional Limit, psi.	Tensile Strength, psi.	Elongation, per cent	Total Extension, $L-L_o$, in.	Total Energy, W, ft-lb.	Critical Velocity, ft. per sec.
1......	70 000	89 500	17.3	0.169	98	95.3
2......	66 000	92 000	10.4	0.204	114	105.4
3......	64 000	88 500	7.6	0.222	121	91.2
4......	61 000	88 000	6.56	0.256	145	103.0
5......	66 000	90 900	5.45	0.266	159	94.6
6......	64 000	90 000	5.2	0.305	181	99.8
7......	67 000	88 500	4.28	0.288	170	92.2
7......	64 000	88 500	5.15	0.352	213	....
8......	65 000	88 000	4.8	0.374	220	104.2
10......	67 000	89 500	3.54	0.345	217	97.5

diameter on the tensile impact properties were machined from bars of cold-rolled S.A.E. 1020 steel $\frac{5}{8}$ in. in diameter. The analysis of this material is as follows:

Carbon, per cent.............	0.23
Silicon, per cent..............	0.27
Manganese, per cent..........	0.73
Phosphorus, per cent.........	0.018
Sulfur, per cent..............	0.034

The diameter of these specimens was 0.300 in. over the gage length. Specimens were produced with gage lengths of 1, 2, 3, 4, 5, 6, 7, 8, and 10 in.

Static stress-strain diagrams were determined for these specimens. Some of the static stress-strain curves are presented in Fig. 1. The properties determined in the static tests are presented in Table I.

The dynamic tests were made at impact velocities of 50, 75, 100, 125, 175, and 200 ft. per sec. The results of these tests are presented in Table II. It has been shown in a previous paper (10) that the ultimate strength may be somewhat greater under dynamic conditions than under static conditions, but at velocities above approximately 25 ft. per sec. it does not seem to increase. The results presented in Table II show that there is essentially no variation of tensile strength with change of gage length.

These results are most evident in the curves of total energy *versus* impact velocity which are plotted in Fig. 2. The effect of impact velocity upon total energy does not seem to be significant for the 1 and 2-in. gage length specimens. The large influence of impact velocity is

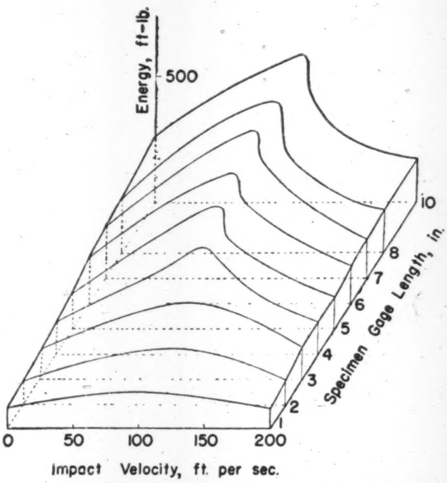

FIG. 2.—Schematic Diagram Showing Influence of Gage Length and Velocity of Impact on Total Energy.

quite evident, however, in tests made with specimens having a 3-in. gage length or greater. The total energy increases with increasing impact velocity. At velocities in the neighborhood of 100 ft. per sec. the value reaches a maximum, and at higher velocities there is a very definite decrease in energy. The important fact is that the *maxima* in the curves of Fig. 2 occur at the same impact velocity, namely, 100 ft. per sec.

The experimentally-determined critical velocity coincides with the computed critical velocity based upon the static stress-strain diagram. In the tests covered by this paper, the diameter of the specimen was constant while the ratio of length to diameter was between 3.3 and 33. It is apparent that in this par-

TABLE II.—RESULTS OF DYNAMIC TESTS ON SPECIMENS OF S.A.E. 1020 COLD-ROLLED STEEL.

Specimen Gage Length, in.	Elongation, per cent	Total Extension, $L-L_0$, in.	Energy, W, ft-lb.	Tensile Strength, psi.	Elongation, per cent	Total Extension, $L-L_0$, in.	Energy, W, ft-lb.	Tensile Strength, psi.	Elongation, per cent	Total Extension, $L-L_0$, in.	Energy, W, ft-lb.	Tensile Strength, psi.	Elongation, per cent	Total Extension, $L-L_0$, in.	Energy, W, ft-lb.	Tensile Strength, psi.
	Static Tests				50 ft. per sec.				75 ft. per sec.				100 ft. per sec.			
1....	17.30	0.169	98	89 500	24.4	0.238	142	110 000	22.80	0.222	117	110 000	30.0	0.299	160	102 000
2....	10.40	0.204	114	92 000	17.7	0.337	206	113 000	15.20	0.297	153	107 000	17.6	0.344	193	105 000
3....	7.60	0.222	121	88 500	13.3	0.390	221	99 000	16.50	0.484	395	121 000	14.3	0.419	323	122 000
													15.4	0.450	280	112 000
4....	6.56	0.256	145	88 000	9.3	0.363	218	106 000	12.40	0.483	302	109 000	14.3	0.557	340	110 000
5....	5.45	0.266	159	90 900	10.3	0.504	327	110 000	10.30	0.504	327	110 000	12.5	0.611	376	109 000
6....	5.20	0.305	181	90 000	9.0	0.525	430	118 000	10.50	0.613	410	110 000	11.4	0.660	500	120 000
					10.5	0.615	375	109 000								
7....	4.28	0.288	170	88 500	10.3	0.705	475	111 000	10.80	0.740	...	......	11.0	0.570	550	120 000
	5.15	0.352	213	88 500					9.30	0.636	375	100 000	11.4	0.592	613	107 000
8....	4.80	0.374	220	88 000	9.0	0.703	450	120 000	8.83	0.688	535	108 000	11.2	0.877	598	110 000
					6.9	0.695	322	112 000								
10....	3.54	0.345	217	89 500	7.3	0.714	420	106 000	8.10	0.792	465	103 000	9.2	0.695	568	108 000
	125 ft. per sec.				150 ft. per sec.				175 ft. per sec.				200 ft. per sec.			
1....	21.2	0.202	126	109 000	25.0	0.240	140	111 000	22.4	0.224	128	115 000	22.4	0.219	131	107 000
2....	20.2	0.395	235	105 000	16.5	0.315	204	120 000	15.7	0.306	183	114 000	14.7	0.287	196	115 000
3....	14.6	0.426	290	116 000	12.7	0.371	197	109 000	11.3	0.332	241	120 000	9.4	0.275	173	109 000
	17.2	0.505	312	110 000												
4....	12.4	0.486	364	112 000	10.0	0.388	261	109 000	6.8	0.226	177	109 000	5.4	0.210	149	108 000
5....	10.5	0.514	320	108 000	8.3	0.404	280	120 000	6.8	0.335	221	112 000	5.9	0.287	121	113 000
6....	9.3	0.546	340	104 000	7.4	0.435	292	105 000	5.7	0.336	218	106 000	4.8	0.284	208	114 000
7....	8.4	0.571	440	115 000	6.7	0.461	...	108 000	4.6	0.318	218	103 000	4.0	0.271	212	111 000
8....	7.5	0.586	435	112 000	4.8	0.372	274	104 000	4.3	0.336	228	103 000	3.02	0.236	178	114 000
10....	5.5	0.539	350	101 000	5.3	0.518	199	103 000	3.2	0.313	194	105 000	....	....	...	......

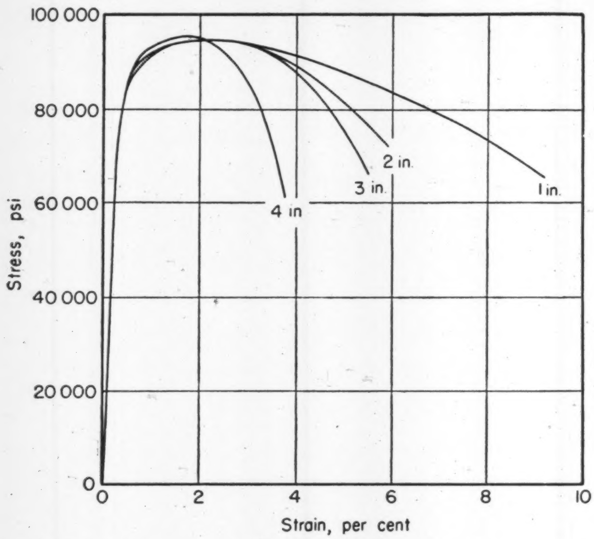

FIG. 3.—Static Stress-Strain Curves for Cold-Rolled Steel (all specimens are 0.15 in. in diameter).

TABLE III.—RESULTS OF STATIC TESTS OF COLD-ROLLED STEEL.
Specimens 0.15 in. in diameter

Gage Length, *L*, in.	Ratio, *L*/*D*	Proportional Limit, psi.	Ultimate Strength, psi.	Elongation, per cent	Energy per Unit Volume, ft-lb. per cu. in.	Theoretical Critical Velocity, ft. per sec.	Reduction of Area, per cent	Rockwell Hardness, B scale
1......	6.7	70 000	94 000	9.0	652	85	53.0	91.8
1......	6.7	71 000	94 300	9.5	628	87	54.0	94.6
2......	13.3	67 000	95 000	7.1	405	90	58.2	92.5
2......	13.3	68 000	95 000	7.2	400	91	56.3	93.7
3......	20.0	68 500	95 000	5.1	394	88	59.1	92.7
3......	20.0	70 000	95 000	4.8	356	90	60.2	92.7
4......	26.6	70 000	95 500	3.5	256	87	63.0	95.0
4......	26.6	71 500	94 500	3.4	254	89	64.1	91.1

ticular instance in which specimens 0.300 in. in diameter were utilized, the gage length of the specimen must be at least 4 in. in order to afford a definite determination of the critical velocity.

A further investigation has been made to determine whether or not geometrically-similar specimens tested at the same impact velocity exhibit identical dynamic tensile properties. For this purpose specimens were manufactured from a cold-rolled steel of the following composition:

Carbon, per cent............	0.19
Manganese, per cent..........	1.03
Phosphorus, per cent.........	0.018
Sulfur, per cent..............	0.029
Silicon, per cent..............	0.34

All specimens were machined to a gage diameter of 0.15 in. Gage lengths of 1, 2, 3, and 4 in. were taken giving a ratio of length to diameter of 6.7, 13.3, 20.0, and 26.6.

The static stress-strain curves for the four gage lengths are presented in Fig. 3. The results of the static and dynamic tests are presented in Tables III and

TABLE IV.—RESULTS OF DYNAMIC TESTS OF COLD-ROLLED STEEL.

Specimen	Impact Velocity, ft. per sec.	Ultimate Strength, psi.	Energy per Unit Volume, ft-lb. per cu. in.	Elongation, per cent	Reduction of Area, per cent	Rockwell Hardness, B Scale
1-IN. GAGE LENGTH, 0.150-IN. DIAMETER						
No. 3	26.0	108 000	1450	17.0	52	92.2
No. 7	49.8	114 000	1230	15.8	50	92.1
No. 5	49.8	106 000	1530	18.6	59	93.0
No. 6	75.0	95 000	1080	17.5	58	91.4
No. 13	101.0	111 000	1190	15.3	68	90.1
No. 4	101.0	102 000	1130	15.7	58	92.4
No. 8	126.0	104 000	805	13.1	60	91.6
No. 9	150.0	104 000	737	9.7	52	92.2
No. 14	150.8	109 000	856	12.6	62	91.5
No. 10	176.0	115 000	907	11.9	51	92.0
No. 11	176.0	110 000	840	12.1	60	92.0
No. 12	201.0	112 000	907	12.5	61	91.7
						91.8 Avg.
2-IN. GAGE LENGTH, 0.150-IN. DIAMETER						
No. 2	25.0	107 000	....	9.0	58	93.8
No. 9	49.5	123 000	1001	10.6	61	92.6
No. 3	50.0	......	....	11.5	58	93.4
No. 4	74.2	112 000	1160	12.7	61	92.8
No. 10	100.0	110 000	1360	13.2	59	92.9
No. 5	125.0	125 000	1000	9.3	61	94.2
No. 6	152.0	115 000	712	7.1	60	93.0
No. 7	181.0	110 000	613	7.7	61	93.8
No. 8	200.0	119 000	575	6.8	61	92.5
						93.2 Avg.
3-IN. GAGE LENGTH, 0.150-IN. DIAMETER						
No. 5	24.7	106 200	642	8.3	58	93.8
No. 6	50.5	97 000	750	9.4	45	96.6
No. 4	75.0	107 000	764	10.7	53	93.9
No. 3	75.1	......	...	8.9	56	93.2
No. 8	99.0	......	...	8.3	53	94.1
No. 7	101.0	105 000	925	9.8	59	93.5
No. 14	125.0	104 000	680	8.7	64	93.1
No. 10	150.0	100 000	453	6.2	59	92.3
No. 11	151.0	100 000	460	7.0	52	93.7
No. 12	176.0	99 000	456	5.6	58	93.5
No. 13	204.0	101 000	382	5.1	62	93.2
						93.7 Avg.
4-IN. GAGE LENGTH, 0.150-IN. DIAMETER						
No. 3	25.3	114 000	615	7.0	58	95.6
No. 4	50.2	113 000	675	7.4	64	94.5
No. 5	75.3	113 000	840	9.3	60	93.8
No. 6	100.0	119 000	765	8.1	62	94.1
No. 8	125.0	112 000	595	5.9	60	91.3
No. 7	126.0	......	...	6.9	57	93.7
No. 9	150.0	124 000	523	4.3	64	95.0
No. 10	178.0	117 000	497	4.1	65	95.1
No. 11	202.0	115 000	447	3.9	55	94.1
						94.1 Avg.

IV, respectively. The curves of percentage elongation *versus* impact velocity are presented in Fig. 4. The location of the maximum in the curve for the 1-in. gage length specimen is difficult to determine. However, a distinct maximum is observed for the 2 and 3-in. specimens and the critical velocity is found to be at about 100 ft. per sec. It is apparent from these results that in order to secure an adequate indication of critical velocity, the ratio of length to diameter must be of the order of 13 or greater.

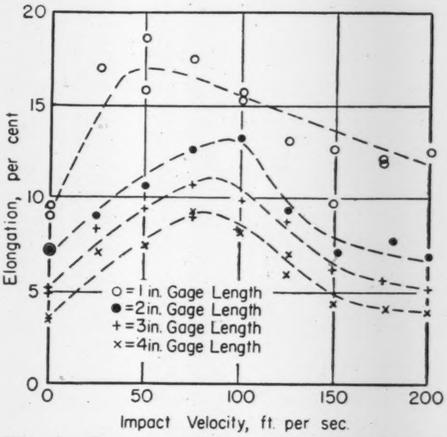

FIG. 4.—Percentage Elongation *versus* Impact Velocity for Cold-Rolled Steel (all specimens are 0.15 in. in diameter. Symbols indicate different gage lengths).

The requirement that tensile impact specimens must have a ratio of length to diameter which is greater than a certain minimum value if the critical velocity is to be clearly defined may be explained by a qualitative argument based upon the theory of the propagation of plastic strain waves. The theory predicts that at impact velocities above the critical velocity the specimen should fail at the moving end with zero elongation. However, the theory does not take the necking phenomenon into account. Experimentally, it is found that necking always occurs. Thus at super critical velocities a neck is formed at the moving end of the specimen, but the remainder of the gage length experiences very little strain in accordance with the theory. To

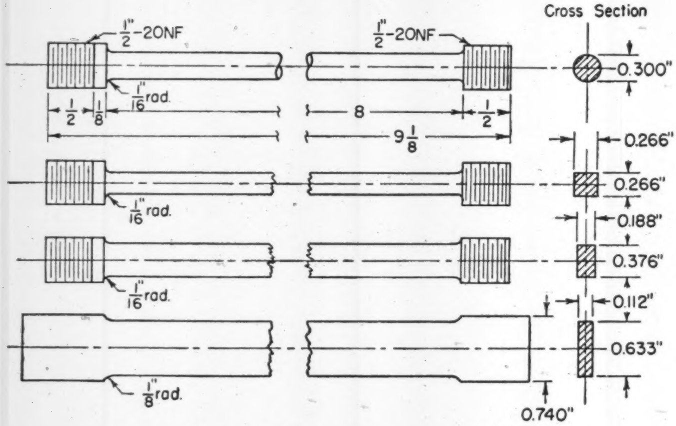

FIG. 5.—Specimens Used in Tests on Annealed Copper.

TABLE V.—RESULTS OF STATIC TESTS OF GEOMETRICALLY SIMILAR SPECIMENS.

Metal	Specimen Size, in.	Specimen	Tensile Strength, psi.	Proportional Limit, psi.	Energy per Unit Volume, ft-lb. per cu. in.	Elongation, per cent	Reduction of Area, per cent	Theoretical Critical Velocity, ft. per sec.	Experimental Critical Velocity, ft. per sec.	Rockwell Hardness
Annealed Copper, Lot I, ½ in.	7 by 0.35	No. 2	29 000	4 000	820	41.4	71	...	...	74.8 F
	7 by 0.35	No. 3	29 500	4 000	845	42.4	70	...	...	73.0 F
	2 by 0.10	No. 11	29 000	4 000	670	32.8	68	...	...	31.5 F
	2 by 0.10	No. 12	29 200	4 000	600	31.5	69	...	...	31.3 F
Annealed Copper, Lot II, 1 in.	16 by 0.6	No. 1	28 400	4 000	756	38.0	69	...	...	38.0 F
	16 by 0.6	No. 2	28 200	4 000	768	39.6	70	...	...	40.0 F
	8 by 0.3	No. 1	29 400	4 000	756	37.6	75	...	...	65.6 F
	8 by 0.3	No. 2	29 300	4 000	670	34.0	70	...	...	64.3 F
	4 by 0.15	No. 1	29 700	4 000	715	34.6	72	...	...	39.5 F
	4 by 0.15	No. 2	29 500	4 000	706	34.8	75	...	...	38.0 F
Cold-Rolled Steel	8 by 0.3	No. 1	85 500	65 000	361	5.4	66	95	100	94.4 B
	8 by 0.3	No. 2	82 500	64 000	442	6.6	66			92.8 B
	4 by 0.15	No. 1	95 500	70 000	256	3.5	63	87	80	95.0 B
	4 by 0.15	No. 2	94 500	71 500	253	3.4	64			91.1 B
Annealed Steel	8 by 0.3	No. 9	66 800	40 800	1070	21.6	68	183	140	73.3 B
	8 by 0.3	No. 10	67 000	40 400	1050	21.4	67			74.2 B
	4 by 0.15	No. 10	73 000	43 000	747	14.1	58	182	150	72.3 B
	4 by 0.15	No. 11	74 000	44 000	773	14.2	55			73.0 B

a first order of approximation the elongation in the neck is independent of impact velocity and is proportional to the specimen diameter, at least for impact velocities of the order of the critical velocity. Therefore, in order to observe the marked decrease in elongation in the unnecked portion of the specimen which characterizes super critical impact velocities by measurements of total elongation, the gage length of the specimen must be much longer than the length of the necked region. Since the length of the necked region is approximately proportional to the specimen diameter, it is clear that a ratio of length to diameter greater than some minimum value must be used if the critical velocity is to be clearly defined.

The influence of size of the test section on the results of tension impact tests was studied by making static and dynamic tension tests on samples of annealed copper, cold-rolled steel and annealed steel. These results are shown in Table V. These data indicate that the size of the test section does not influence the detection of the critical impact velocity.

INFLUENCE OF SHAPE OF CROSS-SECTION OF SPECIMEN ON TENSILE IMPACT RESULTS

Specimens having the cross-sectional shapes indicated in Fig. 5 were employed for the study of the influence of the shape of the cross-section upon tensile impact test results. The cross-sectional area of the specimens was maintained at 0.071 sq. in. and the gage length was maintained at 8 in. The specimens were prepared from $\frac{5}{8}$-in. round bars of hard-drawn copper. The round and square specimens and the specimens of section 0.376 by 0.188 in. were machined from the hard-drawn bar, annealed at 900 F. for $\frac{1}{2}$ hr. and quenched in water. The rectangular specimens, 0.630 by 0.112 in., were prepared in the following manner: (1) bar stock cut to length, annealed at 1200 F. for 1 hr., and water quenched, (2) forged to a flat strip 1 by 1.8 in. in section, (3) annealed at 1200 F. for 1 hr. and water quenched, (4) rolled to 0.112-in. thickness, (5) specimens machined from the rolled strip, and (6) annealed at 850 F. for $\frac{1}{2}$ hr. and water quenched. The temperature of the last anneal was determined experimentally as that temperature which would produce the grain size present in the other section shapes.

TABLE VI.—RESULTS OF STATIC TESTS ON ANNEALED COPPER SPECIMENS.

Specimen Size, in.	Specimen	Tensile Strength, psi.	Proportional Limit, psi.	Energy per Unit Volume, ft-lb. per cu. in.	Elongation, per cent	Reduction of Area, per cent	Rockwell Hardness, F scale
0.3-in. diameter	No. 29	30 000	5000	545	29.0	65	68.3
0.3-in. diameter	No. 30	29 800	5000	718	36.5	64	67.4
0.26 by 0.26 in.	No. 11	30 000	5000	636	33.2	67	59.3
0.26 by 0.26 in.	No. 12	29 600	5000	674	35.0	68	63.6
0.186 by 0.378 in.	No. 23	29 400	5000	672	37.0	71	60.7
0.186 by 0.378 in.	No. 24	29 000	5000	590	32.0	67	62.7
0.112 by 0.633 in.	No. 15	29 800	5000	730	37.4	..	78.0
0.112 by 0.633 in.	No. 16	30 150	5000	740	38.0	..	77.6

Two static tension tests were made on each shape of specimen. Static stress-strain diagrams were determined for these specimens and their differences were not greater than the difference found between curves corresponding to two specimens of the same shape. The results of the static tests are presented in Table VI.

The tensile impact tests were made at impact velocities ranging from 25 to 200 ft. per sec. The results of these tests are given in Table VII. The values of tensile strength, energy required for rupture, and percentage elongation under dynamic conditions are higher than the static values, but the increase is practically the same for all cross-sectional shapes. The difference between results is of the same order of magnitude as the scatter observed for a series of tests on any one of the cross-sectional shapes. While the critical velocity of this material is above 200 ft. per sec., it is to be expected that the cross-sectional shape will not influence its value since the stress-strain diagrams are essentially the same. It can be concluded from this series of tests that within the range of dimensions considered the results of static or dynamic tensile tests are independent of the shape of the cross-section.

TABLE VII.—RESULTS OF DYNAMIC TESTS OF ANNEALED COPPER SPECIMENS OF DIFFERENT SECTION SHAPES.

Cross-Section	Specimen	Impact Velocity, ft. per sec.	Ultimate Strength, psi.	Energy per Unit Volume, ft-lb. per cu. in.	Elongation in 8 in., per cent	Reduction of Area, per cent	Rockwell Hardness, F scale
Circular diameter 0.300 in.	No. 21	25.7	35 000	762	34.9	68	69.2
	No 22	50.6	36 700	1030	42.3	70	70.3
	No. 23	75.1	38 900	1070	45.8	69	65.7
	No. 24	100.0	37 200	1100	46.0	66	66.2
	No. 25	125.5	35 400	1150	50.2	71	66.7
	No. 26	152.0	35 400	1080	49.0	67	68.8
	No. 27	176.3	37 000	992	43.5	68	70.1
	No. 28	202.0	38 000	744	37.7	72	68.1
							68.1 Avg.
Square 0.266 by 0.266 in.	No. 1	25.7	36 200	1140	41.0	65	65.8
	No. 2	50.4	37 800	1250	41.5	66	63.0
	No. 9	74.6	36 200	1180	41.5	72	64.3
	No. 3	74.6	36 400	1305	44.2	69	67.2
	No. 10	101.0	37 000	1180	45.5	71	64.0
	No. 4	104.0	36 000	1060	39.0	72	63.5
	No. 5	124.8	35 600	1180	41.6	69	62.5
	No. 6	150.5	36 000	1150	41.8	70	68.8
	No. 7	175.7	37 000	1225	45.0	73	66.2
	No. 8	202.0	39 000	1210	42.0	70	69.7
							65.5 Avg
Rectangular 0.188 by 0.376 in.	No. 14	24.7	38 000	1085	36.8	65	67.5
	No. 13	25.7	......	....	38.8	67	63.7
	No. 15	49.5	38 400	1135	36.1	71	66.8
	No. 16	74.5	40 000	1400	45.3	68	66.7
	No. 17	100.2	38 000	1190	40.0	69	67.3
	No. 18	124.7	38 500	1245	41.8	65	67.0
	No. 19	150.3	37 400	1270	44.4	70	68.2
	No. 20	173.5	37 600	1380	49.0	73	66.5
	No. 21	199.0	......	....	44.0	70	67.8
	No. 22	201.0	38 000	1230	45.5	71	71.2
							67.3 Avg
Rectangular 0.112 by 0.633 in.	No. 5	25.4	41 200	1030	39.6	..	79.5
	No. 6	25.4	40 000	985	37.0	..	80.8
	No. 7	50.8	40 400	1150	42.0	..	80.4
	No. 17	75.1	41 000	1030	40.7	..	81.4
	No. 9	101.2	40 000	910	34.2	..	81.4
	No. 10	125.7	38 500	1070	41.4	..	80.8
	No. 11	150.5	39 200	900	34.8	..	81.7
							80.7 Avg.

CONCLUSIONS

From the test results presented in this paper it may be concluded that in making tensile impact tests it is necessary to utilize a ratio of length to diameter of specimen of approximately 13 to properly establish the critical velocity. In reviewing the work of previous investigators, it will be noted that in those cases the ratio of length to diameter was less than 13 and hence a critical velocity would not be clearly indicated. The shape of the specimen cross-section does not influence the effect of velocity on the tensile properties of metal within the dimensions studied.

Acknowledgments:

The investigations presented in this paper were conducted at the California Institute of Technology under a contract with the National Defense Research Committee of the Office of Scientific Research and Development. The authors wish to acknowledge the participation of P. E. Duwez in these investigations.

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DISCUSSION

Mr. R. L. Templin[1].—I should like to ask the authors of this well-presented and prepared paper if they have extended their studies to include temperatures other than normal to see if there is any question about a critical velocity at higher or lower temperatures. I think that is a very interesting point in view of the discussions these days on impact testing.

Mr. D. S. Clark (*author*).—We have made some tests at temperatures as low as approximately −75 F. In these tests a critical velocity was observed. The results have not been published.

We observed some other peculiar things which may be related to our rapid-load studies about which I think we will have something to say later.

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[1] Assistant Director of Research and Chief Engineer of Tests, Research Laboratories, Aluminum Company of America, New Kensington, Pa.

SOME EFFECTS OF AUSTENITIC GRAIN SIZE AND METALLURGICAL STRUCTURE ON THE MECHANICAL PROPERTIES OF STEEL*

By G. M. Sinclair[1] and T. J. Dolan[1]

Synopsis

Studies were made of S.A.E. 1045, 2340, and 3140 steels to determine the changes in mechanical properties which accompanied a large increase in the austenitic grain size (from A.S.T.M. No. 10 to No. 3). Each of these steels was heat-treated to produce two general types of microstructure, but all were tempered to a hardness of approximately 100 Rockwell B. It was desired to investigate the influence of austenitic grain size on both the pearlitic and tempered martensitic types of microstructure at the same hardness level; hence, the final hardness was determined primarily by that attainable in the pearlitic steel groups.

For those cases in which the microstructure consisted mainly of *tempered martensite*, it was found in general that (*a*) an increase in the prior austenitic grain size slightly decreased the percentage elongation obtained in static tension tests but did not appreciably affect the yield, tensile, or fatigue strength values for the steel and (*b*) in the transition temperature range, the energy absorbed by V-notch Charpy impact specimens was somewhat greater for the coarse-grained steels; this was felt to be due to incomplete hardening of the same steels when in the fine-grained condition. Heat treatments which produced small amounts of pro-eutectoid ferrite in a matrix of tempered martensite resulted in a steel having mechanical properties which were inferior to those of the same steel more completely hardened (no pro-eutectoid ferrite).

For those cases in which the microstructure was essentially pearlitic, a large increase in the prior austenitic grain size greatly reduced the ductility as measured by percentage elongation and also decreased the energy absorbed in the V-notch Charpy impact test over all ranges of temperature investigated. The yield ratio was increased and the damage produced by a notch in a fatigue test was decreased when the austenitic grain size was coarsened for the pearlitic steels.

The literature of metallurgy has, for a number of years indicated the apparent desirability of maintaining a fine austenitic grain size in our steel microstructures for the purpose of developing greater toughness and strength. The majority of these data have been based upon studies of pearlitic steels, and "toughness" usually has been evaluated by means of the single-blow, notched-bar impact test conducted at room temperature. As is now known, interpretation of the results of the impact test is difficult at best in so far as the design of machine parts is concerned, and impact values obtained for a single temperature must be considered qualitative only.

Recently, a number of investigators (**1**,**2**,**4**,**18**,**19**)[2] have presented data

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Research Associate and Research Professor, respectively, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 615.

indicating that the mechanical properties of steels having a tempered martensitic type of structure are superior to those of a steel having carbides distributed in the form of plates. In general, this has been found to be true for steels having carbon contents below 0.50 per cent and having hardness levels below 50 Rockwell C. There is some evidence (**23, 24, 25, 43**) that at higher carbon and hardness levels, the formation of high residual stresses or microscopic cracks on quenching may become a controlling factor in determining the properties of the steel. Accordingly, under these conditions, the fatigue strength of a steel having a microstructure of tempered martensite (*severe* initial quench) may be appreciably lower than the fatigue strength of the same steel having a supposedly less desirable structure of tempered bainite. If however, the tempered martensite is produced by a martempering and tempering operation (smaller quenching and transformational stresses) its fatigue strength may again become superior to that of bainite (**43**).

The ease with which this desirable martensitic structure can be obtained throughout a steel part is a function of the hardenability of the steel, which, in turn, is primarily determined by the prior austenitic grain size and the alloy content. It would be economical to increase the hardenability of a steel by using a large austenitic grain size, but unfortunately the tendency to form quench cracks or to become embrittled by grain boundary precipitates also increases with grain size (**3**). In view of these opposing effects of grain size on the properties of a quenched-and-tempered steel part, it is conceivable that the most economical use of a given steel may not always be obtained by adhering to the finest grain size.

In ordinary commercial practice, fine-grained quenched-steel parts which have a structure of 100 per cent martensite before tempering are rare. If these same steel parts can be hardened throughout by the use of a larger austenitic grain size, it may be possible to improve some strength characteristics of the part at little cost. Again, there may be occasions in industrial practice where the formation of a coarse austenitic grain size cannot be avoided and it would be desirable to know the extent to which this condition may influence the mechanical properties. In the present paper, data are presented which seem to indicate that under certain conditions no appreciable loss in strength resulted when the austenitic grain size was increased.

A brief survey dealing with the results of several investigations, which were made to study the effects of prior austenitic grain size and metallurgical structure on the mechanical properties of steel, has been included in the Appendix to this paper. It was felt that these investigations showed a lack of agreement on several important points and that they would be of interest for comparison with the results obtained during the present study.

OBJECT AND SCOPE OF THE INVESTIGATION

To help clarify some of the apparently contradictory results found in previous work, an investigation was made to study changes in the mechanical properties of several steels accompanying a large increase in the austenitic grain size. For this purpose three commonly used steels, S.A.E. 1045, 2340 and 3140 were selected and given heat treatments designed to produce two general types of microstructure. The first of these microstructures consisted mainly of tempered martensite while the second was largely pearlite and ferrite. The austenite grain size of the steels tested varied between a maximum of A.S.T.M. No. 3 for the coarsest condition to a minimum of A.S.T.M. No. 10 in the fine condition.[3] All steels were tempered to approximately the *same hardness* (100 Rockwell B) before being tested.

The mechanical properties investigated were those measured by means of the ordinary tension test, the rotating-beam fatigue test and the Charpy notched-beam impact test. The fatigue studies included the determination of *S-N* diagrams for both notched and unnotched specimens and the calculation of fatigue "notch sensitivity" (6) from these data. V-notch Charpy impact values were determined for the various steels and microstructures over a range of temperatures, including the range of "transition" from a ductile to a brittle type of fracture. Impact-temperature curves plotted from the test data were compared on the basis of microstructure and grain size in a separate graph for each steel.

TABLE I.—CHEMICAL ANALYSES OF STEELS TESTED.

Steel	Analyses, per cent						
	C	Mn	P	S	Si	Ni	Cr
S.A.E. 1045[a]	0.44	0.74	0.018	0.032	0.20	...	...
S.A.E. 1045[b]	0.44	0.75	0.014	0.021	0.21	...	...
S.A.E. 3140	0.37	0.75	0.017	0.030	0.178	1.33	0.65
S.A.E. 2340	0.40	0.74	0.019	0.020	0.28	3.48	...

[a] Used for series 9, 10 and 16.
[b] Used for series 1 and 2.

As a related problem, the mechanical properties of a "mixed" microstructure of tempered martensite with small amounts of pro-eutectoid ferrite at the grain boundaries were also determined. These tests were limited to the S.A.E. 1045 and 2340 steels and only a coarse (A.S.T.M. No. 3 to 4) austenite grain size was investigated. The term "grain size" as used in this paper, unless otherwise indicated, is intended to refer to the *prior austenitic* grain size.

PREPARATION OF SPECIMENS AND METHOD OF TESTING

Heat Treatments of Specimens:

The steels were obtained as hot-rolled round bars $\frac{7}{8}$ in. in diameter and had the chemical compositions listed in Table I. The S.A.E. 3140 and 2340 steels were taken from the same heats as those used in the preceding investigation by T. J. Dolan and C. S. Yen (1); however, the plain carbon steels were from different heats and varied slightly in chemical composition as indicated in Table I.

The bars were cut into segments $9\frac{5}{8}$ in. long, and turned to $\frac{5}{8}$-in. diameter preparatory to heat treatment. Those segments of S.A.E. 1045 and 3140 which were to be made into fatigue specimens were further reduced to $\frac{1}{2}$-in. diameter so that approximately the same cooling rates would be obtained in the center of these specimens as were used by Dolan and Yen (1).

Details of the various heat treatments used in this investigation and of the metallurgical structures which resulted are presented in Tables II and III, and in Figs. 18(*a*) through 23(*b*) respectively. Each heat treatment for a particular steel was assigned a series number.[4] The odd-numbered groups (series 1, 3, 5, 9, 11, and 13) were considered to be rapidly quenched; while the even numbered groups (series 2, 4, 6T, 10, 12, and 14) were relatively slowly cooled.

The austenitizing procedures used for most series were very similar. The cold stock was placed in an electric furnace kept at the austenitizing temperature and was then held for the time indicated

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[3] Standard Classification of Austenite Grain Sizes in Steels, 1949 Book of A.S.T.M. Standards, Part 1, p. 1271.

[4] For convenience in comparing the results, it should be remembered that the test series designated by small numbers (1 to 8) were fine-grained, whereas those designated by large numbers (9 to 16) were large grained.

TABLE II.—HEAT TREATMENT AND MECHANICAL PROPERTIES OF STEELS HAVING A TEMPERED MARTENSITIC TYPE OF MICROSTRUCTURE.

S.A.E. Steel	Series	Time at Temperature, hr.	Austenitized, deg. Fahr.	Quench	Temper 1 hr., deg. Fahr.	A.S.T.M. Grain Size	Rockwell Hardness B scale	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength (0.2 per cent offset), psi.	Tensile Strength, psi.	Yield Ratio, $\frac{\text{Yield Strength}}{\text{Tensile Strength}}$	Charpy Impact Test			Endurance Limit, psi.		K_f[c]	Endurance Ratio		Notch Sensitivity Index, q[d]
													At 70F.	At −42F.	Transition Temperature, deg. Fahr.	Unnotched	Notched		Unnotched	Notched	
No. 1045.....	1	1/2	1520	Water	1210	6	99.7	22.5	62.8	83 800	104 700	0.80	28.7	8.2	>+70	61 500	26 000	2.37	0.59	0.25	0.72
No. 1045.....	9	1/2	1800	Water	1150	3	98.8	17.6	56.4	74 100	106 500	0.70	78.0	2.0	+65	62 000	23 500	2.64	0.58	0.22	0.86
Ratio $\frac{\text{Values for Series 1}}{\text{Values for Series 9}}$							1.01	1.28	1.11	1.13	0.98	1.15				0.99	1.11	0.90	1.01	1.13	0.83
No. 2340.....	5	1/2	1450	Oil	1200	10	101.9	23.7	67.0	96 000	115 700	0.83	92.5	83.6	−95	71 000	30 000	2.37	0.61	0.26	0.72
No. 2340.....	11	1/2	1850	Oil	1200	4	100.5	23.0	64.7	91 000	114 400	0.80	95	89	−100	70 000	28 000	2.50	0.61	0.25	0.79
Ratio $\frac{\text{Values for Series 5}}{\text{Values for Series 11}}$							1.01	1.03	1.04	1.06	1.01	1.04				1.02	1.07	0.95	1.00	1.06	0.91
No. 3140.....	3	1/2	1520	Oil	1300	10	101.3	25.5	60.7	87 200	108 200	0.81	120.2	108.2	−45	65 500	28 000	2.34	0.61	0.26	0.71
No. 3140.....	13	4	1970	Oil	1300	3	100.6	18.1	64.3	89 900	112 900	0.80	120	108	−65	68 000	32 000	2.12	0.60	0.28	0.59
Ratio $\frac{\text{Values for Series 3}}{\text{Values for Series 13}}$							1.01	1.41	0.95	0.97	0.96	1.01				0.96	0.88	1.10	1.00	0.92	1.20
No. 2340.....	15	[a]	[a]	[a]	[a]	4	98.1	19.7	61.2	81 800	108 500	0.75	95	82	−100	65 000	24 000	2.71	0.60	0.22	0.90
No. 1045.....	16	[b]	[b]	[b]	[b]	3	98.5	15.5	43.2	86 000	112 800	0.76	57	12	+16	57 000	23 000	2.48	0.51	0.20	0.78

[a] Heat treatment for Series 15; austenitized 1/2 hr. 1850 F. quenched in salt 30 min. at 1100 F. water quenched, temper. 1 hr. 1200 F.

[b] Heat treatment for Series 16; austenitized 1/2 hr. 1800 F. quenched in salt 100 sec. at 1200 F. water quenched, temper. 1 hr. 1150 F.

[c] K_f = strength reduction factor = $\frac{\text{endurance limit of unnotched specimen}}{\text{endurance limit of notched specimen}}$.

[d] $q = \frac{K_f - 1}{K_t - 1}$ where K_t = the theoretical stress concentration factor.

TABLE III.—HEAT TREATMENT AND MECHANICAL PROPERTIES FOR STEELS HAVING A PEARLITIC TYPE OF MICROSTRUCTURE.

S.A.E. Steel	Series	Time at Temperature, hr.	Austenitized, deg. Fahr.	Quench	Temper 1 hr., deg. Fahr.	A.S.T.M. Grain Size	Rockwell Hardness, B Scale	Elongation in 2 in., per cent	Reduction of Area, per cent	Yield Strength (0.2 per cent offset), psi.	Tensile Strength, psi.	Yield Ratio, $\frac{\text{Yield Strength}}{\text{Tensile Strength}}$	Charpy Impact Test			Endurance Limit, psi.		K_f^a	Endurance Ratio		Notch Sensitivity Index, q^b
													70F.	−42F.	Transition Temperature, deg. Fahr.	Unnotched	Notched		Unnotched	Notched	
No. 1045.....	2	1/2	1520	Oil	1050	6	102.3	21.3	57.3	81 900	119 900	0.68	17.4	5.2	>+70	67 000	27 000	2.48	0.56	0.22	0.79
No. 1045.....	10	1/2	1800	Oil	1050	3	99.5	14.3	48.9	80 900	114 800	0.71	5.0	3.0	190	61 000	25 000	2.44	0.53	0.22	0.76
Ratio $\frac{\text{Values for Series 2}}{\text{Values for Series 10}}$							1.03	1.49	1.17	1.01	1.04	0.97				1.10	1.08	1.02	1.05	1.00	1.04
No. 2340.....	6T	1/2	1450	Air	700	10	101.2	22.3	58.0	78 800	118 900	0.66	60.1	17.3	+65	67 000	21 000	3.19	0.56	0.18	1.15
No. 2340.....	12	1/2	1850	Air	700	4	101.0	12.5	46.3	82 100	121 700	0.68	19.0	14.0	140	66 000	23 000	2.87	0.54	0.19	0.98
Ratio $\frac{\text{Values for Series 6T}}{\text{Values for Series 12}}$							1.00	1.78	1.25	0.96	0.98	0.98				1.02	0.91	1.11	1.03	0.95	1.17
No. 3140.....	4	1/2	1520	Air	1050	10	99.4	24.7	68.0	74 600	109 000	0.68	81.9	17.7	+65	62 500	22 000	2.84	0.57	0.20	0.97
No. 3140.....	14	4	1970	Air	1140	3	99.7	17.0	57.4	81 000	107 900	0.75	28.0	14.0	90	61 000	26 000	2.35	0.57	0.24	0.71
Ratio $\frac{\text{Values for Series 4}}{\text{Values for Series 14}}$							1.00	1.45	1.19	0.92	1.01	0.90				1.02	0.85	1.21	1.00	0.83	1.37

[a] K_f = strength reduction factor = $\frac{\text{endurance limit of unnotched specimen}}{\text{endurance limit of notched specimen}}$.

[b] $q = \frac{K_f - 1}{K_t - 1}$, where K_t = the theoretical stress concentration factor.

in Table II. For most series, no protective atmosphere was used, and the segments were quenched directly from the austenitizing temperature. Tempering was delayed until 2 to 3 hr. after quenching. The S.A.E. 3140 steel used for series 13 and 14 was packed in a charcoal-filled box and held at 1970 F. for 4 hr. These segments were also quenched directly from the austenitizing temperature.

The slight amount of surface decarburization which occurred during the above treatments was readily removed during machining of the specimens and was considered negligible in the test results.

Series 15 and 16, S.A.E. 2340 and 1045 steels, respectively, were given special heat treatments designed to produce a "mixed" structure consisting mainly of martensite with small percentages of high-temperature transformation products at the grain boundaries. The heat treatment for series 15 (S.A.E. 2340) consisted of austenitizing as for series 11 and 12 and then quenching in a nitrate salt bath at 1100 ± 5 F. The specimens were held in the salt bath for 30 min. and then water quenched to transform the remaining austenite. The temperature of the bath was constantly checked by means of a thermocouple. Specimens were quenched in the salt in small batches of three or four at a time to prevent overheating of the bath.

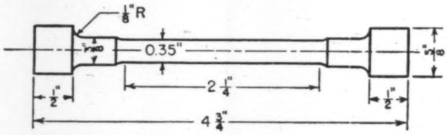
Series 16 (S.A.E. 1045) was austenitized in the same manner as series 9 and 10 and was then quenched in the nitrate salt bath for 100 sec. at 1200 F. Specimens were water quenched immediately after being removed from the salt bath.

Hollomon and Jaffe's temperability curves (39) were used in exploratory tests, and from these a tempering procedure was determined for each series so as to give a final hardness of approximately 100 Rockwell B.

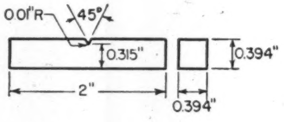
Machining and Mechanical Testing of Specimens:

The dimensions of the specimens machined from each group or heat-treated series of specimens are shown in Fig. 1. Each $9\frac{3}{8}$-in. segment could be machined into three fatigue specimens, four impact specimens, or one tension and two impact specimens. Very light cuts were used in all cases during the final machining operations to reduce the amount of disturbed surface metal.

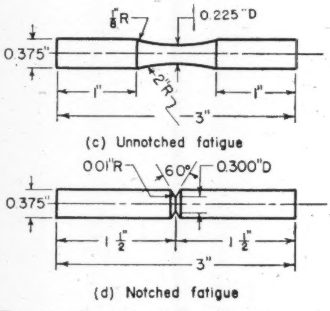
The tension specimens (Fig. 1(*a*)) were given a final polish with No. 1 grit emery paper, and the diameter at the mid-section was reduced gradually to 0.004 in. less than the rest of the specimen to aid in obtaining a fracture near the center of the gage length. It should be noted that the gage length of 2 in.



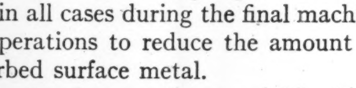
(a) Static tension



(b) Charpy impact bending



(c) Unnotched fatigue



(d) Notched fatigue

FIG. 1.—Details of Test Specimens.

was about 6 diameters in these specimens instead of the more commonly used $L = 4D$. Three tension specimens were machined from each heat-treated series and were tested in an Amsler Universal Testing Machine. The yield strength and tensile strength were determined from the complete stress-strain diagrams.

The unnotched fatigue specimens (Fig. 1(*c*)) were given a standard polish using three successively finer grades of emery paper. The scratches left by each polishing operation were made to cross the scratches left by the previous operation. The final finish was with 00 emery paper and oil with the direction of scratches being parallel to the longitudinal axis of the specimen. The minimum diameter of these specimens was measured to the nearest 0.0001 in. by means of a Pratt & Whitney "Supermicrometer."

The notch of the "notched" flexual fatigue specimens (Fig. 1(*d*)) was machined using a carefully honed tool. The accuracy of contour of this tool was checked against a template at a magnification of 50× by micro-projection. No attempt was made to polish the root of the notch in the specimen, but it was examined at 50× for accuracy of contour and to insure against deep scratches and tears.

The fatigue tests were performed at a speed of 7000 to 8000 rpm. on Krouse fatigue machines of the rotating cantilever-beam type. All stresses were computed by means of the ordinary flexure formula:

$$S = \frac{Mc}{I}$$

where:

S = the maximum fiber stress,

M = the moment applied,

c = the distance from the outer fiber to the neutral axis, and

I = the moment of inertia of the critical section.

The stresses for the notched specimens were computed on the basis of the minimum cross-sectional area without regard to the stress concentration caused by the notch.

V-notch Charpy impact specimens were machined to the dimensions in Fig. 1(*b*); the notch was cut by milling with a special formed cutter. The specimens of each heat-treated series were heated or cooled in liquid baths to the desired temperature and then broken in a standard impact machine of 223 ft. lb. capacity. A mixture of dry ice and petroleum ether was used to cool specimens in the range from room temperature to −100 F. For temperatures ranging from −100 F. to −200 F. the petroleum ether bath was set in a second container which had been packed with asbestos fiber, and small amounts of liquid air were poured in on the asbestos layer.[5] A small oil bath was used to heat those specimens which were tested above room temperature. Each specimen was held at the bath temperature for a minimum of 10 min. before being broken. It was assumed that the change in temperature of the specimen, during transfer from the bath to the testing machine, would be negligible.

Determination of Austenitic Grain Size:

Photomicrographs which show the prior austenitic grain size of the steels resulting from the various austenitizing treatments used in this study are presented in Figs. 15, 16, and 17. It should be noted here that *none* of the microstructures shown in these illustrations were from specimens tested mechanically and that the photographs are intended merely to give an indication of the size of austenite grain produced in each given steel by the austenitizing treatment used for the test specimens.

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[5] Precautions were taken to insure that no ether or other oxidizable material would come in contact with the liquid air (which results in a ***dangerous explosive mixture***).

Since all of the steels were of hypoeutectoid composition, the *large* austenite grain sizes were outlined by means of precipitated ferrite resulting from a treatment involving slow cooling. The plain carbon steel (S.A.E. 1045 in Fig. 15(*a*)) was cooled in air from the austenitizing temperature and the two alloy steels (Figs. 16(*a*) and 17(*a*)) were furnace cooled. Grain sizes produced by the low austenitizing temperatures used by Dolan and Yen (1) were so fine that the ordinary methods of delineating grain boundaries (controlled cooling, gradient quench, martensitic etch, etc.) were unsatisfactory for photographic reproduction.

Photographs of the fine-grained steels (Figs. 15(*b*), 16(*b*), and 17(*b*)) appearing in this report were obtained from samples prepared by a special technique developed by E. J. Eckel and S. Paprocki (44). Briefly, the method consisted of polishing the surfaces of two samples of the steel to be checked for grain size; these surfaces were coated with tin and then placed in contact with one another to exclude oxygen. The steel samples were then austenitized at the time and temperature of interest. Shortly before the austenitizing time had elapsed, a piece of tinned copper was placed in the furnace and heated to the steel temperature. The tinned surfaces of the steel specimens were then separated and immediately placed in contact with the prepared surface of the copper plate. Approximately $\frac{1}{2}$ hr. was allowed for the molten bronze (produced by the solution of copper in the molten tin) to penetrate the austenite grain boundaries of the steel samples which were then water quenched. The prepared surface was given a *light* metallographic polish (just through the bronze layer) and etched with 4 per cent picral solution; the martensite appeared light while the grain boundaries were outlined in the darker etching bronze. The size of the austenite grains delineated in this manner was checked visually against that obtained by the gradient quench and martensitic etch methods and appeared to be the same.

In Tables II and III are listed the A.S.T.M. grain sizes corresponding to the various heat treatments used in this investigation. These values were determined according to the A.S.T.M. procedure previously noted and were checked by photographing three or four representative areas of the specimen at 100×, and making a count of the number of grains contained in a 4 by 4-in. square ruled on each photograph; one-half of the number of grains which were intersected by the boundary line were included in the total count. From these data the average number of grains per square inch at 100× was found and the grain size was calculated from the formula:

average number of grains per square inch at a magnification of $100\times = 2^{N-1}$

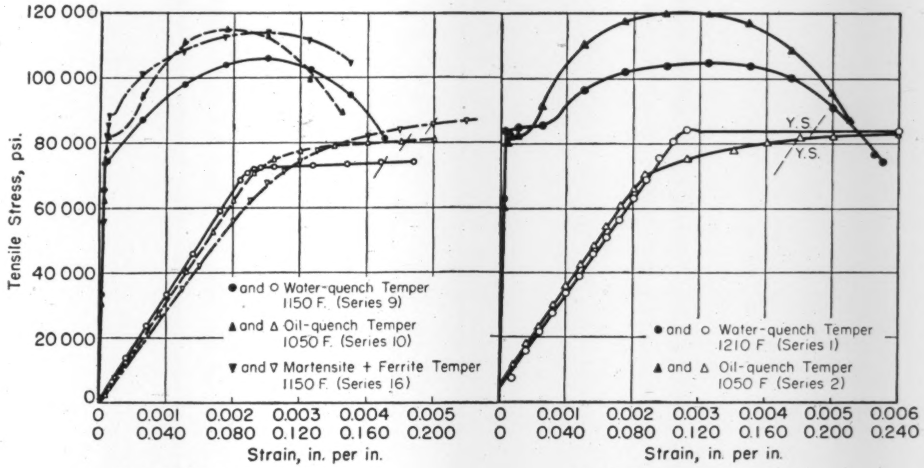
where N is the grain size number.

Microstructure of Heat-Treated Steels:

Metallographic examinations of the various microstructures were made on disks cut transversely from the midlength of the specimen blanks. Photomicrographs of the metallurgical structures of series 9 through 14 are shown both before and after tempering in Figs. 18 through 23. In Figs. 24 and 25 are shown the "mixed" structures of tempered martensite and ferrite obtained by series 15 and 16 heat treatments, respectively. The two photomicrographs presented in Fig. 26 were taken on a longitudinal section of an unnotched fatigue specimen of series 16 which was stopped before it had failed completely. The path of the fatigue crack through the mixed structure of tempered martensite and ferrite is clearly defined.

RESULTS AND DISCUSSION

A majority of the test results have been summarized graphically in Fig. 14. Those series shown in the upper portion of Fig. 14 (above the heavy black line) had microstructures of the tempered martensitic type, while those series below this line had structures consisting mainly of ferrite and pearlite. The bar graphs for the fine-grained series of each steel were filled in by crosshatching to simplify comparison with the graphs of the coarse-grained series which were left open. Results for the two special series, Nos. 15 and 16, were included in the "tempered martensitic" microstructure section. A majority of the effects of a large change in grain size on the static tensile and fatigue characteristics of the various steels and microstructures may be compared more readily from this chart than from consideration of the numerical data given in Tables II and III. It was felt, however, that a more complete representation of the changes in impact behavior of the steels was shown in the impact-temperature curves presented in Figs. 11, 12, and 13 than that indicated by the values of energy absorption at two arbitrary temperatures as shown in Fig. 14.

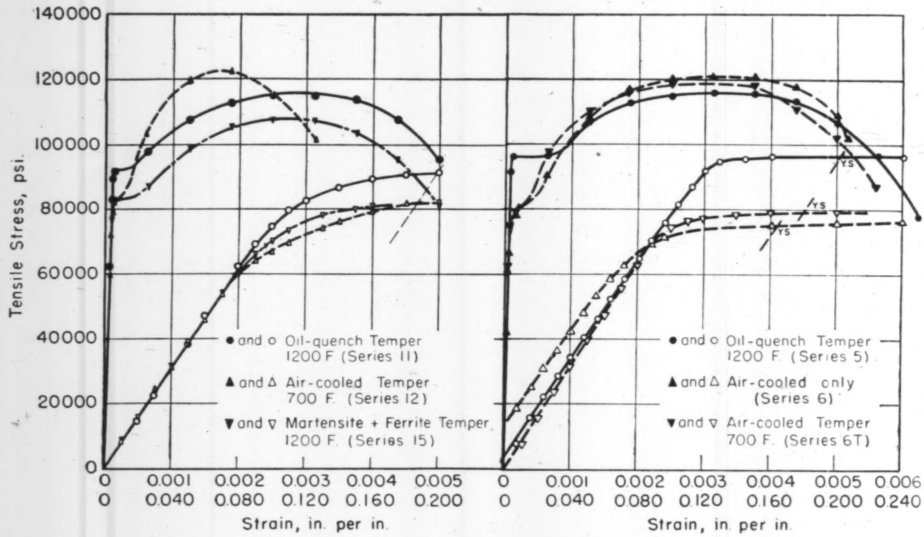


(a) A.S.T.M. Grain Size No. 3. **(b) A.S.T.M. Grain Size No. 6.**

FIG. 2.—Tensile Stress-Strain Curves S.A.E. 1045 Steel.

Note.—Use upper strain scale for open symbols which show lower portions of stress-strain curves.

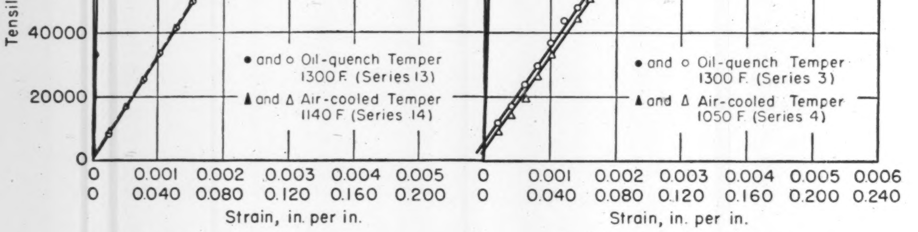
Because of the variety of experiments involved in this study, the discussion of results has been subdivided in accordance with the type of test. Sub-sections on static tension, fatigue and impact tests are presented in that order. Although all steels were tempered to approximately the same hardness before testing, the tensile strengths varied somewhat from one heat-treated group to another. In view of this, the various steels were also compared on the basis of their ratios of endurance limit and yield strength to tensile strength, as well as on the basis of equal hardness.



(*a*) **A.S.T.M. Grain Size No. 4.** (*b*) **A.S.T.M. Grain Size No. 10.**

FIG. 3.—Tensile Stress-Strain Curves S.A.E. 2340 Steel.

Note.—Use upper strain scale for open symbols which show lower portions of stress-strain curves.



(*a*) **A.S.T.M. Grain Size No. 3.** (*b*) **A.S.T.M. Grain Size No. 10.**

FIG. 4.—Tensile Stress-Strain Curves S.A.E. 3140 Steel.

Note.—Use upper strain scale for open symbols which show lower portions of stress-strain curves.

Static Tensile Characteristics:

For Approximately the Same Hardness.—Representative samples of the ordinary stress-strain curves obtained from the various heat treatments of each steel are shown in Figs. 2(*a*) through 4(*b*). For each steel, the left-hand diagram shows typical curves obtained when the austenitic grain size was large, while the right-hand diagram shows the curves that resulted when the grain size was small. The lower portions of each

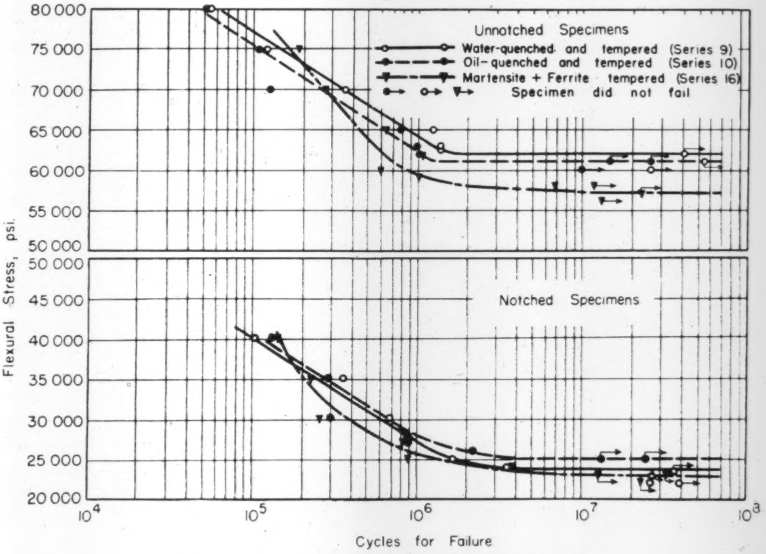


FIG. 5.—*S-N* Curves for S.A.E. 1045 Steel.

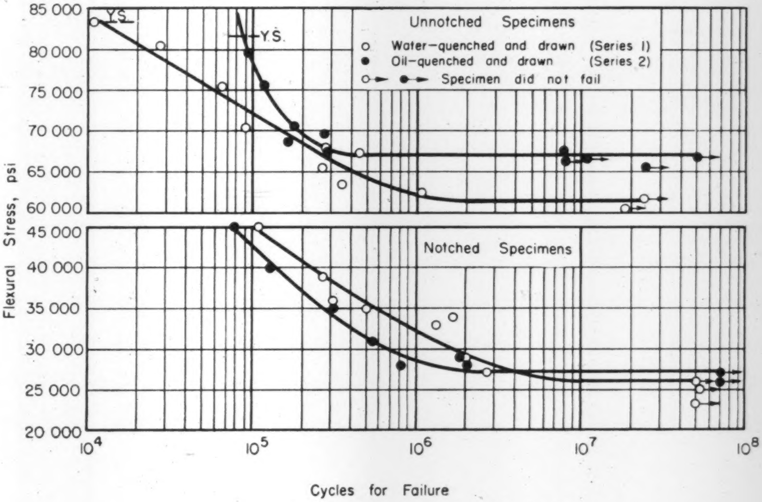


FIG. 6.—*S-N* Curves for S.A.E. 1045 Steel.

curve have also been replotted to an enlarged strain scale on the same diagram to emphasize characteristic differences in strain behavior at the lower stresses.

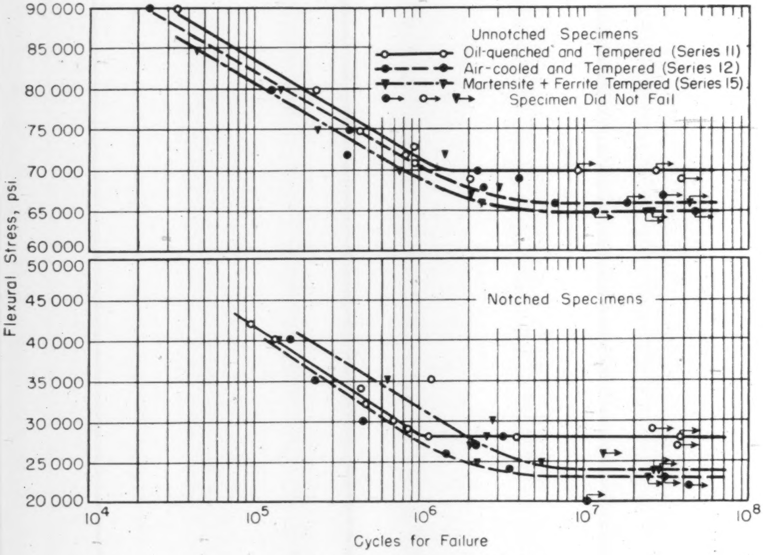


FIG. 7.—*S-N* Curves for S.A.E. 2340 Steel.

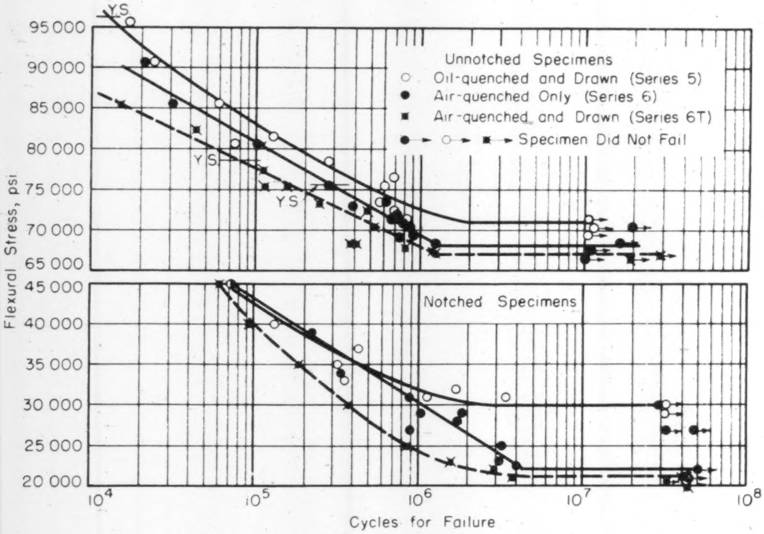


FIG. 8.—*S-N* Curves for S.A.E. 2340 Steel.

In general it was noted from a study of Fig. 14 that the ductility of a steel (as measured by percentage elongation and reduction of area) decreased as the austenitic grain size became larger. This effect was pronounced when the microstructure was pearlitic, and was observed to a lesser extent when the microstructure was of a tempered martensitic type. The influence of a coarse austenitic grain size on the ductility of the S.A.E. 2340 steel was striking in that a coarsening of the grain size from A.S.T.M. No. 10 to No. 4 lowered the average elongation from a value of 23.3 per cent to 12.5 per cent when the structure was pearlitic; the same change in grain size only lowered the elongation value from 23.7 to 23.0 per cent for a tempered martensitic type structure. Similar trends were shown by the other steels. In general these observations were in agreement with the data of Delbart and Potazkim (5), J. H. Hollomon (2), and R. E. Lorentz (9).

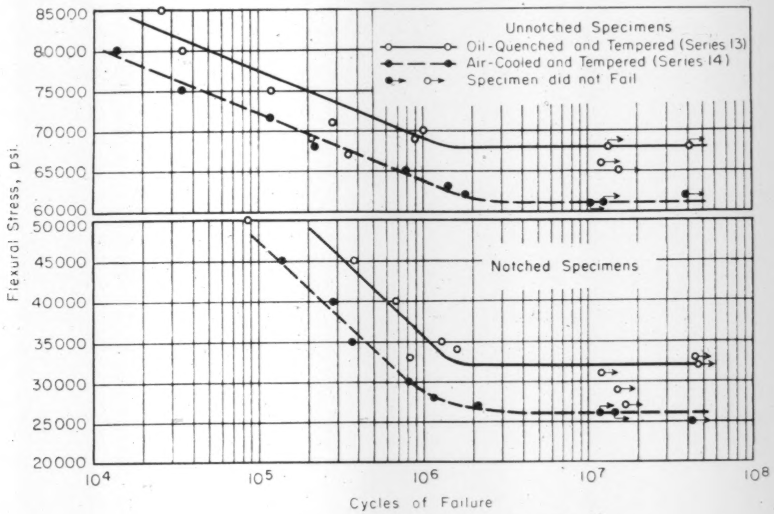


Fig. 9.—*S-N* Curves for S.A.E. 3140 Steel.

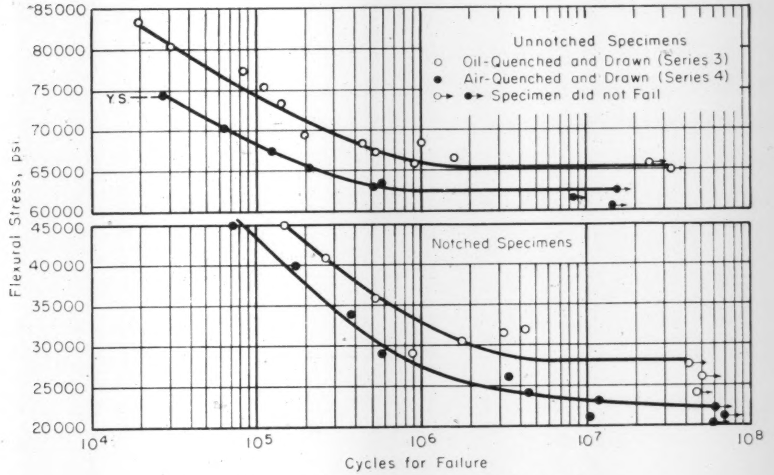


Fig. 10—*S-N* Curves for S.A.E. 3140 Steel.

Tension specimens of S.A.E. 2340 and 1045 steels which were heat treated so as to produce mixed microstructures of ferrite in a matrix of tempered martensite (series 15 and 16) were found to have less ductility than comparable specimens of the same steels without grain boundary ferrite (series 11 and 9).

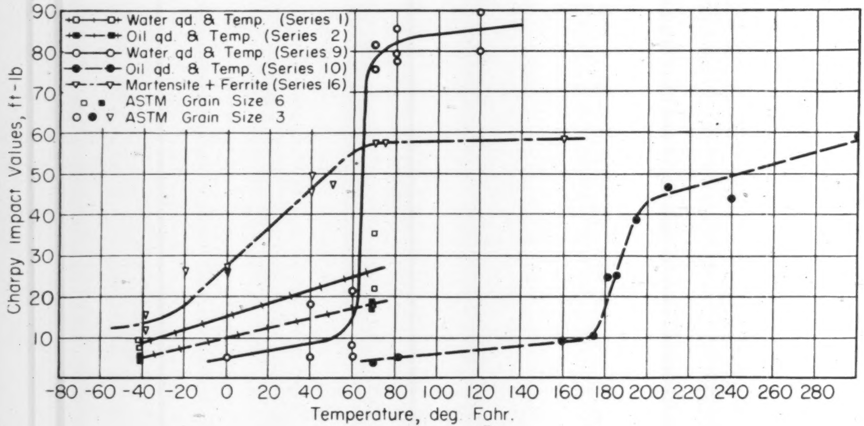


FIG. 11.—Effect of Temperature on Energy Absorbed in the Charpy Test (S.A.E. 1045 Steel).

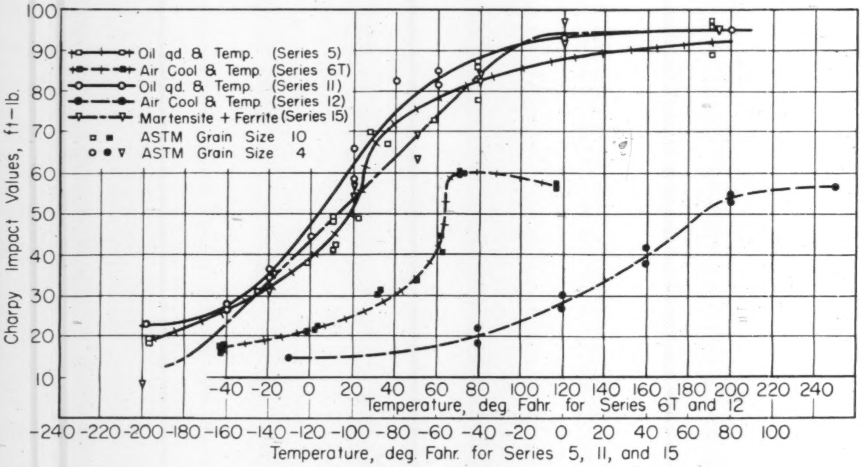


FIG. 12.—Effect of Temperature on Energy Absorbed in the Charpy Test (S.A.E. 2340 Steel).

As may be surmised from the trend in elongation values shown by the steels, a large change in grain size altered the shape of the ordinary stress-strain curves. The change in shape of the "plastic" portion of the curves was most noticeable for the pearlitic steels which showed the greatest loss in ductility in the coarse-grained condition. For example, this may be illustrated by the typical curves shown for the air-cooled S.A.E. 2340 steel (series 12 and 6T) in Figs. 3(*a*) and 3(*b*), respectively. Although the tensile and yield strengths of these two series did not differ appreciably, the strain at maximum load for the coarse-grained steel (series 12) was something less than 0.08 in. per in., whereas that for the fine-grained steel (series 6T) was approximately 0.12 in. per in. By a similar comparison of the stress-strain curves for the other pearlitic series, it was observed that an increase in the prior austenite grain size was accompanied by an increase in the apparent rate of strain hardening of the steel. Stress-strain curves for the steels having a tempered martensitic type of structure exhibited similar tendencies but to a limited extent. In general, specimens of the fine-grained series were able to distribute the strains more uniformly over the entire gage length and achieved greater elongation before local necking began.

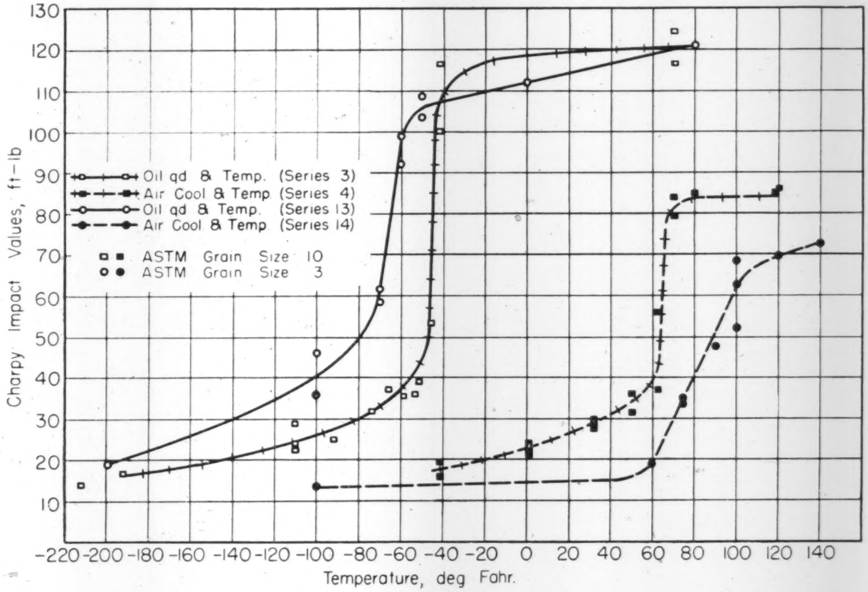


FIG. 13.—Effect of Temperature on Energy Absorbed in the Charpy Test (S.A.E. 3140 Steel).

The "plastic" portion of the curves for the two steels that had a mixed microstructure of ferrite and tempered martensite (series 15 and 16 in Figs. 3(*a*) and 2(*a*), respectively) showed a stress-strain behavior similar to that of the more completely martensitic series. It should be noted, however, that series 16 began to deviate from the "elastic" portion of the curve at lower stresses (lower proportional limit) than the other two series shown in Fig. 2(*a*). It was felt that this behavior at low stress was due to slight yielding of the ferrite network in the microstructure of series 16 (shown in Fig. 25).

When the various steels were compared on the basis of equal hardness, no consistent differences in the effects of grain size on yield strength or tensile strength were apparent.

The Effect of Grain Size on the Ratio of Yield Strength to Tensile Strength.—It was felt that a more equitable comparison of the influence of grain size on the strength characteristics could be made on the basis of the dimensionless ratio

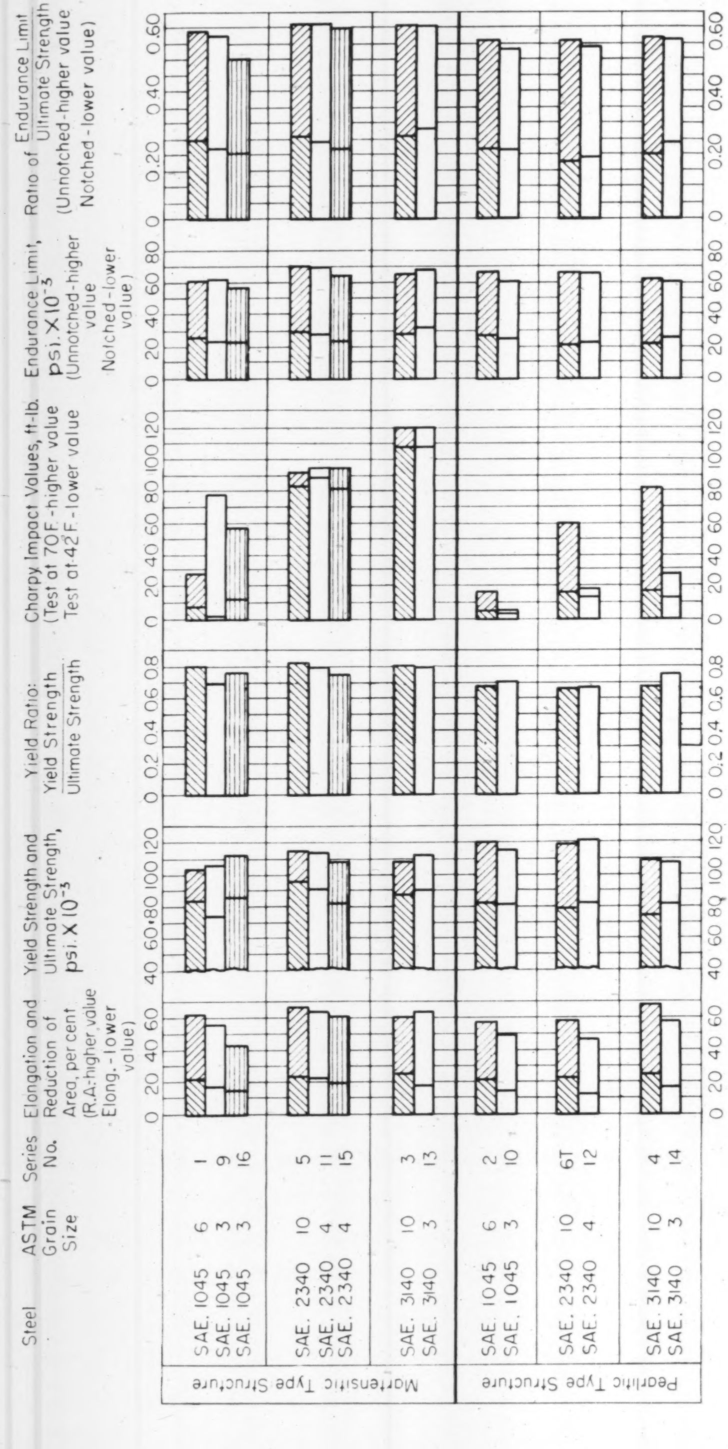
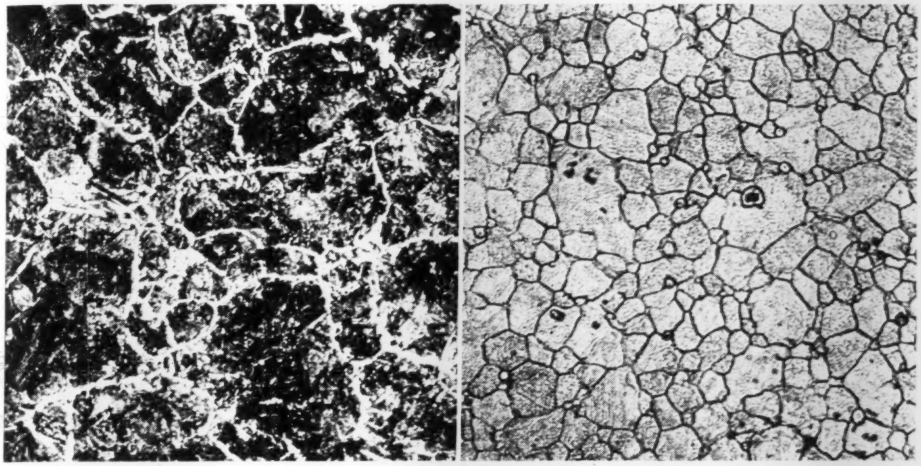


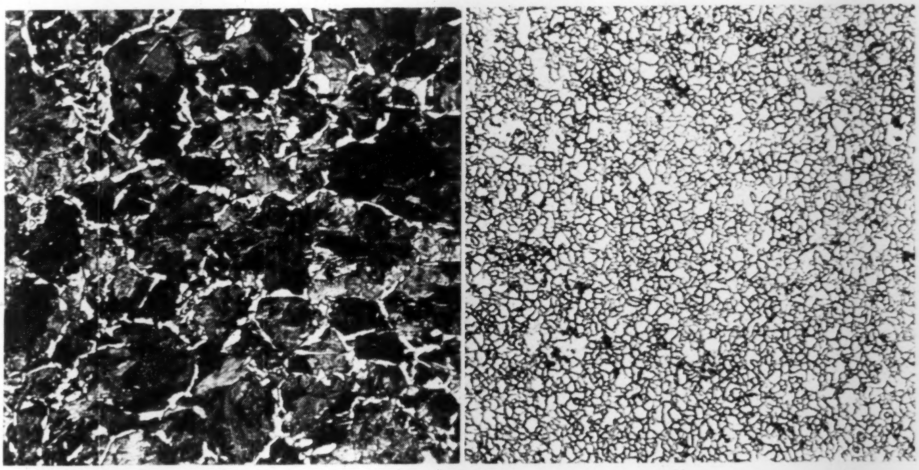
FIG. 14.—Summary of Results of Tests.



(*a*) Heated 1 hr. at 1800 F. (austenitizing treatment of series 9, 10, and 16) and cooled in air. Former austenite grains delineated by Ferrite. A.S.T.M. Grain Size No. 3. Etched in 2 per cent Nital.

(*b*) Heated 1 hr. at 1520 F. (austenitizing treatment of series 1 and 2) Eckel-Paprocki bronze diffusion method used to outline grains. A.S.T.M. Grain Size No. 6. Etched in 4 per cent Picral.

FIG. 15.—Determination of Austenitic Grain Size for S.A.E. 1045 Steels (×100).

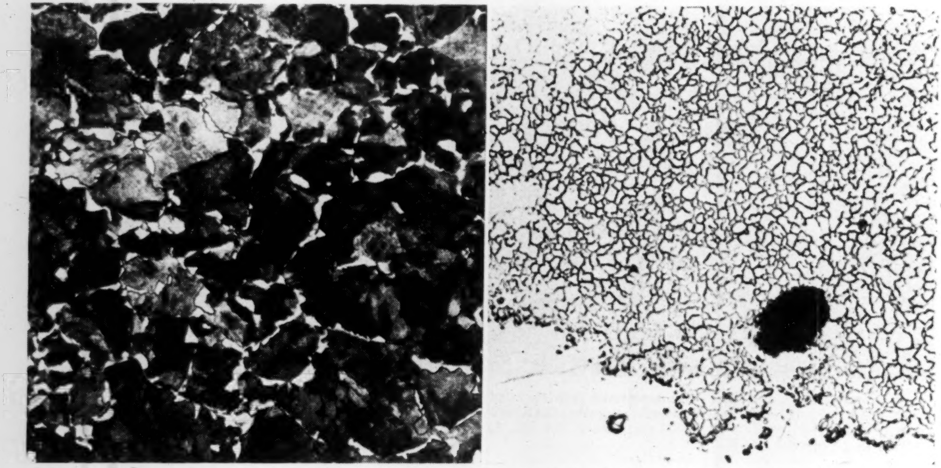


(*a*) Heated 1 hr. at 1850 F. (austenitizing treatment of series 11, 12, and 15) Former austenite grains delineated by ferrite. A.S.T.M. Grain Size No. 4. Etched in 2 per cent Nital.

(*b*) Heated 1 hr. at 1450 F. (austenitizing treatment of series 5, 6, and 6T) Eckel-Paprocki bronze diffusion method used to outline grains. A.S.T.M. Grain Size No. 10. Etched in 4 per cent Picral.

FIG. 16.—Determination of Austenitic Grain Size for S.A.E. 2340 Steel (×100).

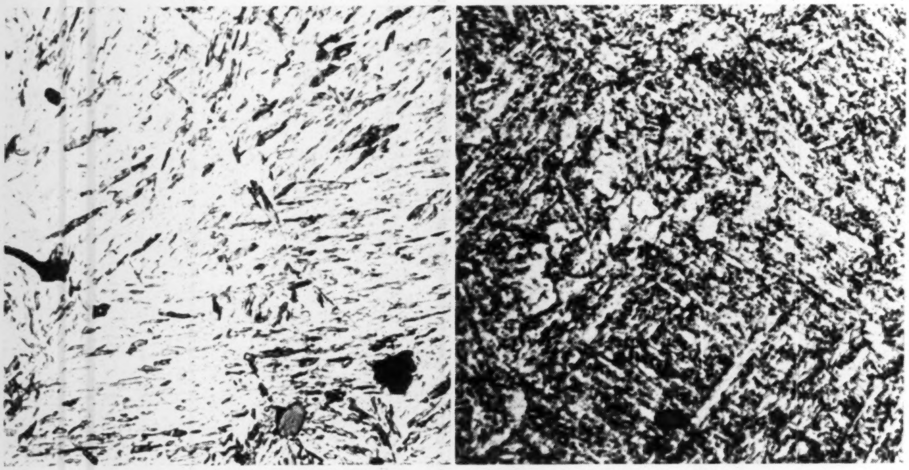
of yield strength to ultimate strength (i.e. the yield ratio—see Fig. 14). In general, it was found that the yield ratio of the series which had microstructures of a tempered martensitic type were higher than the yield ratios of the same steels in the pearlitic condition, irrespective of the prior austenitic grain size.



(*a*) Heated 4 hr. at 1970 F. (austenitizing treatment of series 13 and 14, furnace cooled). Former austenite grains delineated by ferrite. A.S.T.M. Grain Size No. 3. Etched in 2 per cent Nital.

(*b*) Heated 1 hr. at 1520 F. (austenitizing treatment of series 3 and 4) Eckel-Paprocki bronze diffusion method used to outline grains. A.S.T.M. Grain Size No. 10. Etched in 4 per cent Picral.

FIG. 17.—Determination of Austenitic Grain Size for S.A.E. 3140 Steel (×100).

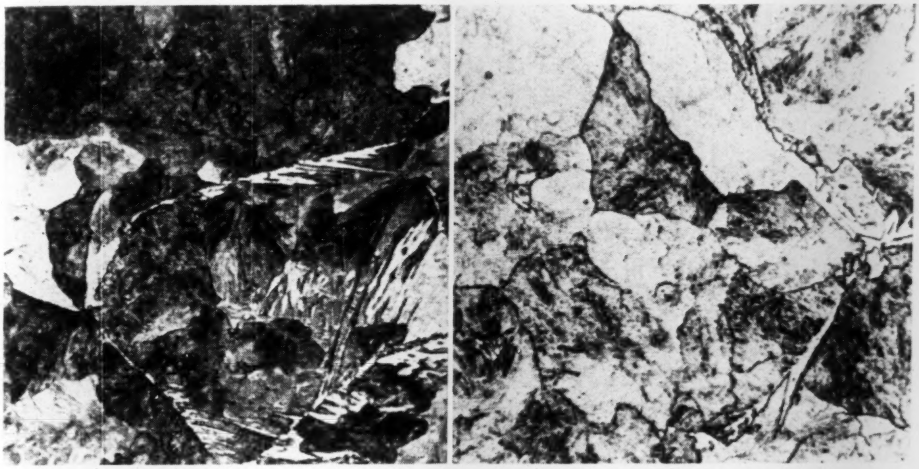


(*a*) (Untempered). Water quenched from 1800 F. Structure consists primarily of martensite with small amounts of unresolved pearlite (primary troostite) at the grain boundaries.

(*b*) (Tested). Water quenched from 1800 F. and tempered 1 hr. at 1150 F. Structure primarily tempered martensite with traces of primary troostite.

FIG. 18.—Microstructure of Series 9 (S.A.E. 1045 Steel). Etched in 2 per cent Nital (×500).

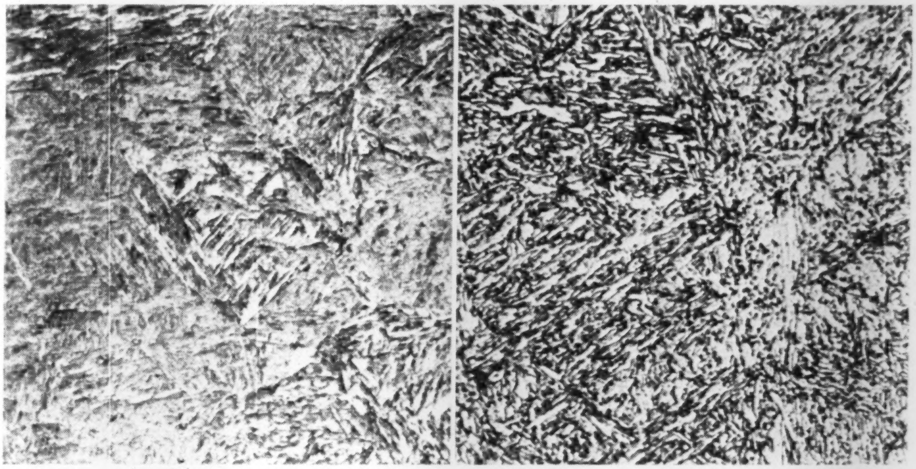
Among the series which had "tempered martensitic" structures, the S.A.E. 2340 and 3140 steels (oil-quenched) showed a very slight decrease in yield ratio when the grain size was coarse. For the plain carbon steel (S.A.E. 1045), an increase in grain size was accompanied by a more pronounced decrease in yield ratio from 0.80 for series 1 (A.S.T.M. grain size No. 6) to 0.70 for series 9 (A.S.T.M. grain size No. 3). It was believed that this decrease in yield ratio for series 9 might possibly be due



(*a*) (Untempered). Oil quenched from 1800 F. Structure mainly fine pearlite with some small areas of martensite. Small amounts of pro-eutectoid ferrite appear at prior austenite grain boundaries.

(*b*) (Tested). Oil quenched from 1800 F. and tempered 1 hr. at 1050 F. Structure tempered pearlite with small quantities of tempered martensite and pro-eutectoid ferrite.

FIG. 19.—Microstructure of Series 10 (S.A.E. 1045 Steel). Etched in 2 per cent Nital (×500).

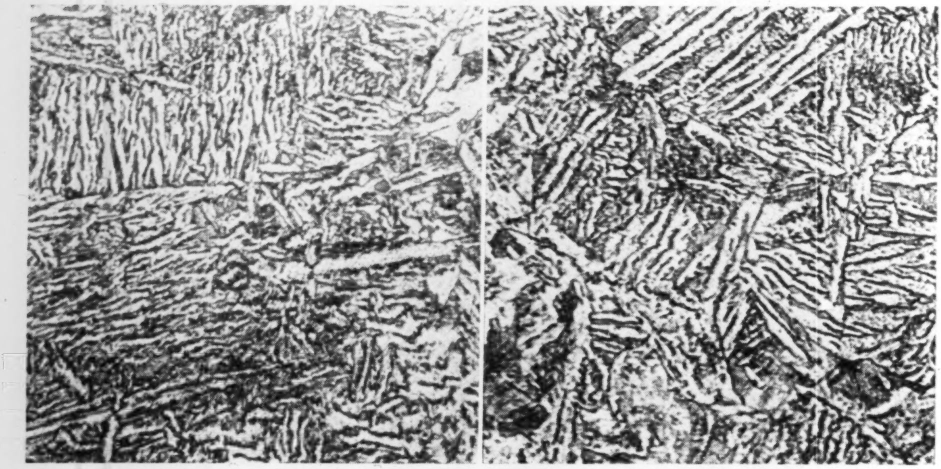


(*a*) (Untempered). Oil quenched from 1850 F. Structure primarily martensite with some evidence of retained austenite.

(*b*) (Tested). Oil quenched from 1850 F., tempered 1 hr. at 1200 F. Structure is tempered martensite.

FIG. 20.—Microstructure of Series 11 (S.A.E. 2340 Steel). Etched in 2 per cent Nital (×500).

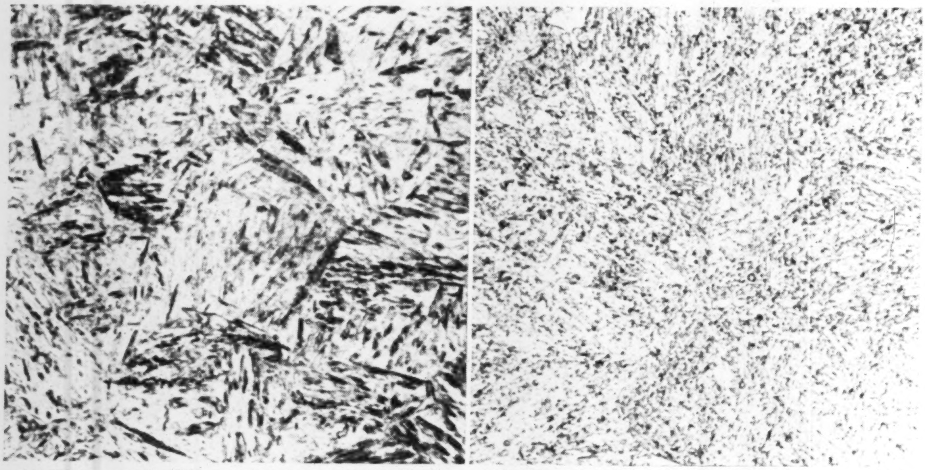
to the presence of minute quench cracks, which could have formed during the severe water quench from the high austenitizing temperature. This belief gained some support from the fact that series 16, which had the same austenite grain size and chemical composition as series 9, was water quenched from 1200 F. and had a yield ratio of 0.76 as compared to 0.70 for series 9. However, no evidence of quench-cracking was observed in microscopic examination of several small



(*a*) (Untempered). Cooled in still air from 1850 F. Structure fine pearlite and ferrite in Widmanstatten pattern.

(*b*) (Tested). Air cooled from 1850 F. and tempered 1 hr. at 700 F. Structure ferrite (Widmanstatten) and tempered pearlite.

FIG. 21.—Microstructure of Series 12 (S.A.E. 2340 Steel). Etched in 2 per cent Nital (×500).



(*a*) (Untempered). Oil quenched from 1970 F. Structure martensite with some evidence of retained austenite.

(*b*) (Tested). Oil quenched from 1970 F., tempered 1 hr. at 1300 F. Structure essentially tempered martensite.

FIG. 22.—Microstructure of Series 13 (S.A.E. 3140 Steel). Etched in 2 per cent Nital (×500).

samples of the "as-quenched" specimens examined at a magnification of 500×.

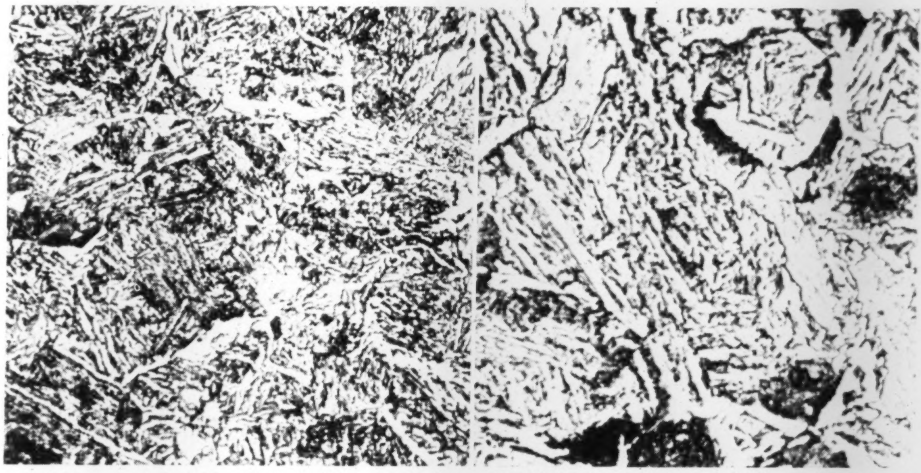
For those treatments which resulted in a microstructure composed mainly of pearlite and ferrite, the coarse-grained series exhibited slightly higher yield ratios than the fine-grained series in all cases.

Effect of Grain Size on Fatigue Characteristics:

Values for the endurance limit of notched and unnotched fatigue specimens of each series were determined from the *S-N* curves presented in Figs. 5 through 10. The highest stress at which several specimens would run 30 to 35 million cycles without failure was designated as the endurance limit. Since the tensile strengths of the steels varied slightly from one series to another, the ratio of the endurance limit to tensile strength was calculated to facilitate a comparison of the fatigue properties presented in Tables II and III. As a relative measure of the resistance of the steel to the effect of notches in a fatigue test, the "notch sensitivity" index, q, discussed by Peterson (6) was computed for all steels according to the equation:

$$q = \frac{K_f - 1}{K_t - 1}$$

From a consideration of the analysis of Neuber (41), the theoretical stress concentration factor, K_t, was found to be 2.9 for the 60-deg. V-notch employed on the fatigue specimens. Peterson (6) has shown that the sensitivity index, q, is not a material constant but it does serve as a rough measure of the sensitivity of the various heat-treated groups to the damaging effects of a notch in fatigue.


(*a*) (Untempered). Cooled in still air from 1970 F. Structure fine pearlite plus ferrite (Widmanstatten).

(*b*) (Tested). Air cooled from 1970 F., tempered 1 hr. at 1140 F. Structure pearlite plus ferrite, some spheroidization of carbides apparent at higher magnification.

FIG. 23.—Microstructure of Series 14 (S.A.E. 3140 Steel). Etched in 2 per cent Nital (×500).

Fatigue Characteristics of Steels at Approximately Equal Hardness.—The fatigue characteristics of the various heat-treat series, when compared on the basis of equal hardness, showed no consistent trends directly attributable to the prior austenitic grain size. The endurance limits for the unnotched specimens which had a pearlitic microstructure were found to vary in approximately the same proportion as their tensile strengths. This also appeared true for the "tempered martensitic" series with the exception of series 16 (S.A.E. 1045 steel) which had a "mixed" microstructure of ferrite in a tempered martensitic matrix. Although this series had a higher tensile strength than either of the comparable series 1 and 9 (Fig. 14), its endurance limit was the lowest of these three groups. It was noted that the values of endurance limit for the unnotched specimens were usually somewhat higher than would be predicted by the commonly used approximation of one-half the tensile strength. In general, the slope of the finite life portion of the *S-N* diagrams was greater for the notched specimens than it was for the unnotched.

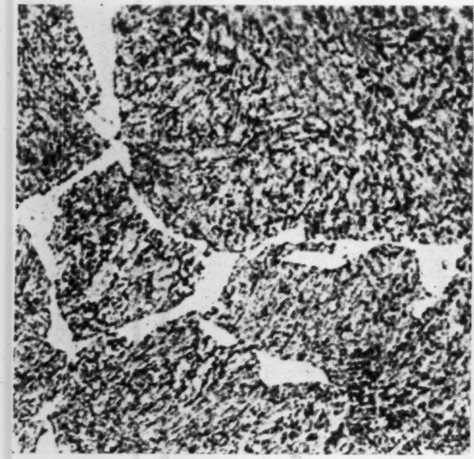


FIG. 24.—Microstructure of Series 15 (S.A.E. 2340 Steel).

Austenitized 1850 F.; quenched in salt bath 1100 F., held one half hour then water quenched. Tempered 1200 F. Structure tempered martensite with ferrite at grain boundaries. Etched in 2 per cent Nital (×500).

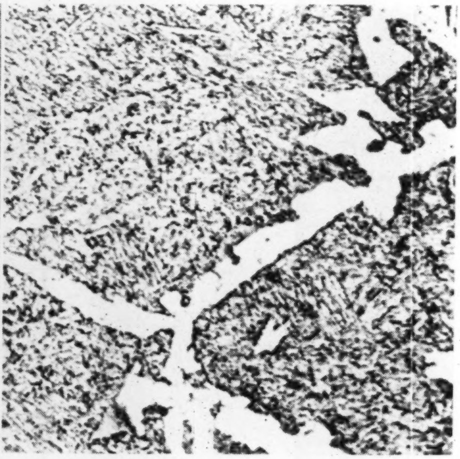


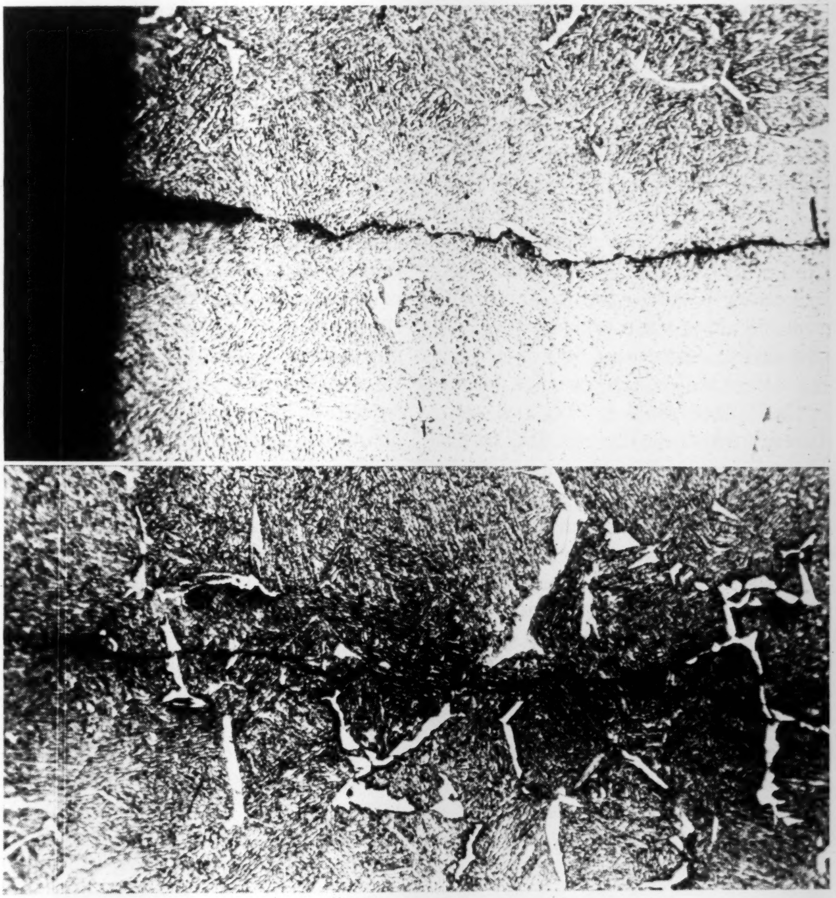
FIG. 25.—Microstructure of Series 16 (S.A.E. 1045 Steel).

Austenitized 1800 F.; quenched in salt bath 1200 F., held 100 sec. and then water quenched. Tempered 1150 F. Structure tempered martensite with ferrite and a few scattered pearlitic areas at grain boundaries. Etched in 2 per cent Nital (×500).

Apparent Influence of Grain Size on Endurance Ratio.—The effect of grain size on the relative endurance limit was studied by comparing the ratios of endurance limit to ultimate strength (endurance ratio) for each series. These values, shown in Tables II and III, were summarized graphically in the last column of Fig. 14. It was interesting to note that, normally, those steels which had the highest yield ratio also had the highest endurance ratio. This has previously been reported by Dolan and Yen (1) and Hollomon (2). For those series which had a structure mainly of tempered martensite, a coarse grain size did not appear to have seriously affected the endurance ratios for either the notched or the unnotched specimens. Endurance ratio values for series 9 were lowered slightly when the grain size was coarsened, which was consistent with its decreased yield ratio.

The special groups that had a structure of ferrite in a matrix of tempered martensite (series 15 and 16) do show distinctly lower endurance ratios and slightly greater notch-sensitivity in fatigue than the corresponding coarse-grained tempered martensite (series 11 and 9 respectively). This seemed to indicate that the small weak areas of precipitated ferrite in series 15 and 16 were able to function as "metallurgical notches" and thus lower the fatigue resistance of the steel. It was interesting to note from Fig. 26 that, once the fatigue crack had penetrated a short distance into the specimen, it did not necessarily follow the ferrite network at the prior austenitic grain boundaries.

With an increase in austenite grain size, the *pearlitic* steels showed a slightly greater resistance to the effect of notches as indicated by the strength reduction factor, K_f and the sensitivity index, q, presented in Table III.



(*a*) (*Upper*) Outer edge of fatigue specimen.
(*b*) (*Lower*) Midlength of fatigue crack.

FIG. 26.—Path of Fatigue Crack in "Mixed" Microstructure of Tempered Martensite and Ferrite (Series 16) Etched in 2 per cent Nital (×250).

Possible Explanation for the Effects of Microstructure on Some Tensile and Fatigue Characteristics of Steel.—From the static tensile data of Tables II and III it has been noted that, at approximately equal hardness, the yield ratios of "tempered martensitic" specimens were higher than the yield ratios of pearlitic specimens of the same steel. When the austenitic grain size was large, the differences between yield ratios were somewhat reduced but were still clearly apparent. A possible explanation for this behavior may be based on Gensamer's theory of strength of metallic aggregates.

It has been shown by Gensamer, *et al.* (**4**) that the resistance of a steel to tensile deformation is proportional to the logarithm of the mean straight path through the ferrite phase. For a fully pearlitic structure, the mean straight path refers to the average distance from one cementite plate to the adjacent one as measured along a random straight line. When pearlite forms during continuous cooling of austenite, the pearlite formation takes place over a *range* of temperatures, and since the spacing of the cementite plates is a function of the reaction temperature (**4, 45**) the distances between adjacent cementite plates are varied over a corresponding range. When a specimen having a non-uniform distribution of carbides is loaded in tension, the regions of coarse pearlite (formed at the higher temperatures) should be the first to deform plastically. As the ferrite of these coarse pearlitic areas becomes strengthened through strain hardening, further plastic deformation will be transferred to other regions which have slightly shorter mean ferrite paths. For a given increment of plastic strain it appears probable that the corresponding increase in flow stress will be greater for steels having a wide variation in the spacing of cementite plates than for steels having a more uniform distribution of carbides. The tempering of martensite results in a precipitation of carbide particles throughout the ferrite matrix, in which the *average* distance between particles does not vary appreciably from one area of the steel to another. It thus appears that for a given tensile strength, the stress required to produce a *small* plastic strain in specimens of tempered martensite should be greater than the stress required to produce the same small strain in pearlitic specimens. From this it follows that when sufficiently small offsets are used to determine yield strength, the yield ratio of tempered martensitic steel will be greater than the yield ratio of pearlitic steel.

According to this line of reasoning, the effects of austenitic grain size on the tensile characteristics would depend mainly upon how this grain size affects the final distribution of carbides in the structure. In the case of tempered martensite, distribution of carbide particles would be changed little if at all by a change in austenitic grain size so that only small changes in tensile characteristics would be expected. For a steel air-cooled to produce a pearlitic structure, coarsening the austenitic grain size would cause a relatively large change in the tensile characteristics because the pearlite reaction would require a longer time to go to completion and thus would occur over a greater range in temperature (resulting in wide variation in the carbide spacing).

The existence of "weak" areas of coarse pearlite in the air-cooled steels may help to explain why the endurance limits of the pearlitic specimens were generally inferior to those of tempered martensitic structure. Since fatigue is a localized phenomenon, it is conceivable that a crack initiated in one of the weak areas would have little trouble in propagating itself through the remainder of the specimen. This argument gains some support from the fact that when "weak" areas of ferrite were present in a matrix of tempered martensite (see series 15 and 16) the *endurance ratio* was appreciably lowered.

Effect of Grain Size on Impact Characteristics:

Curves showing the effect of temperature on the energy absorbed by the V-notch Charpy impact specimens are presented in Figs. 11, 12, and 13 respectively. The experimental points from which the average curves were drawn for each series were included in these figures.

In general, the steels having structures of the tempered martensitic type had much lower transition temperatures and absorbed more energy above this temperature than did the same steel with a pearlitic structure. These changes in impact behavior which accompanied a change in the general type of microstructure were considerably greater than differences attributable to a change in austenitic grain size alone.

For the steels having *pearlitic* structures, an increase in austenitic grain size shifted the "transition" zone to a higher temperature and gave lower values of energy absorption over all ranges of temperature investigated. It was interesting to note in Fig. 13 that had the pearlitic specimens of S.A.E. 3140 steel (series 4 and 14) been tested at room temperature only (70 F.) the resulting impact values would have indicated that the fine-grained series 4 was almost three times as "tough" as series 14. Obviously such results obtained at a single temperature cannot adequately describe the impact superiority of one steel as compared with another.

An increase in the grain size of the "*tempered martensitic*" steels gave rather surprising results. It was found that the coarse-grained series had transition temperatures slightly *lower* than the corresponding fine-grained series. X-ray diffraction patterns of the coarse-grained series of S.A.E. 2340 steel indicated that small amounts of austenite were retained after quenching, but this was not held to be a significant factor in the shift of temperature of transition (3). A possible explanation for this behavior appeared to be incomplete hardening at the center of the fine-grained specimens. Some traces of very fine pearlite were observed microscopically in the center of the as-quenched S.A.E. 1045 specimens. The gain in hardenability which accompanied an increase in austenitic grain size was apparently sufficient to allow thorough hardening and resulted in a slight improvement of the impact properties.

SUMMARY AND CONCLUSIONS

In a laboratory investigation conducted to study the effects of a large increase in prior austenitic grain size on the mechanical properties of steels, three commonly used steels, S.A.E. 1045, 2340, and 3140, were selected and given heat treatments designed to produce two general types of microstructure. The first of these microstructures consisted mainly of tempered martensite, while the second was largely pearlite and ferrite. The austenite grain size of the steels tested varied between a maximum of A.S.T.M. No. 3 for the coarse-grained condition to a minimum of A.S.T.M. No. 10 in the fine-grained condition. As a related problem, the mechanical properties were also determined for a "mixed" microstructure of tempered martensite with small amounts of proeutectoid ferrite at the prior austenite grain boundaries. All steels were tempered to approximately the *same hardness* (100 Rockwell B scale) before being tested.

The mechanical properties investigated were those measured by means of the ordinary tension test, the rotating-beam fatigue test and the Charpy notched-beam impact test. Under the conditions imposed during this investigation the test data appear to justify the following conclusions:

1. In general, the steels which had a tempered martensitic type of structure exhibited mechanical properties superior to those of the same steels with a microstructure of pearlite, irrespective of the prior austenite grain size.

2. With the exception of a decrease in ductility in static tension, the mechanical properties of the steels having a tempered martensitic type of structure were not appreciably affected by an increase in the austenitic grain size.

3. An increase in the prior austenitic grain size of the steels heat treated to produce a microstructure of *pearlite* resulted in:

   (*a*) An increase in the yield ratio in tension.

   (*b*) A decrease in the fatigue notch sensitivity of the steel.

   (*c*) A decrease in the amount of energy absorbed by V-notch Charpy impact specimens, over the entire range of temperatures investigated.

4. Small amounts of pro-eutectoid ferrite in a matrix of tempered martensite resulted in fatigue and static tensile properties inferior to those of the same steel in a tempered martensitic condition.

5. When the prior austenitic grain size was increased in the steels having pearlitic microstructure, the fatigue "notch sensitivity" decreased but the Charpy impact "notch sensitivity" increased. The commonly accepted test methods of evaluating present-day concepts of notch sensitivity lead to basically different trends and cannot be used interchangeably in predicting fatigue and impact behavior.

Acknowledgments:

This investigation has been conducted by members of the staff of the Department of Theoretical and Applied Mechanics of which F. B. Seely is Head. The study was a part of the work of the Engineering Experiment Station of the University of Illinois in cooperation with the Office of Naval Research, United States Navy, under Contract N6-ori-71, Task Order IV. Acknowledgment is also due T. M. Heckler and C. S. Schenk, who aided in the preparation of illustrations.

APPENDIX

The literature concerning the effects of changes in metallurgical structure on the mechanical properties of steel has been particularly voluminous; an extensive review would be too lengthy for presentation in this paper. It was felt, however, that the following investigations would be of particular interest for comparison with the results obtained from the experimental program conducted as a part of the present study.

The data and techniques used in the investigation by T. J. Dolan and C. S. Yen (1) were used extensively in planning the experimental program and in presenting the results in the main body of this paper. They studied the extent to which changes in metallurgical structure affected the fatigue strength and notch-sensitivity of several steels in which the prior austenite grain size was very fine. Duplicate tests were made of three steels in a drastically quenched and tempered condition (tempered martensitic type of structure) and in a slowly quenched and tempered condition (pearlitic structure) all drawn to the *same hardness level.* In general, the steels rapidly cooled in quenching exhibited higher yield ratios, slightly higher endurance limits and less notch-sensitivity in fatigue than the same steels slowly quenched and tempered. The steels consisting mainly of tempered martensite had much lower transition temperatures in the Charpy impact test and also showed higher energy absorption above the transition temperature than the same steels with tempered pearlite structures.

S. A. Herres and C. H. Lorig (3) made

some interesting observations on the effect of prior austenite grain size on the mechanical properties of steel. They suggested that the increase in hardenability, accompanying an increase in austenitic grain size, should improve both the *strength* and toughness under conditions where the same steel in a fine-grained condition would not be hardened throughout in quenching. They concluded that in the absence of grain boundary precipitates fully-hardened steels exhibited "no tendency" for impact resistance to decrease as grain size was increased. (In a discussion of the paper, Jaffe, Hurlich and Wallace (3) disagreed with this conclusion.)

Herres and Lorig also proposed that the brittleness encountered in drastically quenched steels with carbon content greater than 0.30 per cent may be caused by *large transformational stresses* and *micro-cracks* in the martensite grains. Such stresses and micro-cracks increase with severity of quench and are accentuated by a depression of the M_s point and by increased austenitic grain size.

Recently, M. Baeyertz, W. F. Craig, Jr., and J. P. Sheehan (50) have reported that the austenite grain size at the instant of quenching appears to have no significant influence on the notched-bar impact properties of tempered martensite. They suggested that reports of such influence may have been caused by a hitherto unrecognized "brittleness factor".

In a study of 0.1 per cent and 0.4 per cent carbon steel, G. Delbart and R. Potaszkim (5) reported that in the annealed condition steels having either fine or coarse austenitic grain size exhibited substantially the same tensile strength. When both were quenched and tempered, the fine-grained material was slightly inferior in strength. In the annealed condition, elongations were "identical," but in the quenched and tempered condition the fine-grained steel gave a slightly greater elongation. With regard to reduction of area, the fine-grained steel was superior, especially when quenched and tempered.

R. E. Peterson (6) stated that a fillet, notch, or other form of stress concentration introduces a steep stress gradient at the surface as compared with a conventional unnotched fatigue specimen. If the steel is coarse-grained,[6] the stress may drop 25 per cent in crossing a single grain at the surface where the stress gradient is high. The fatigue behavior in such a case would be expected to be different from that obtained in unnotched specimens for which the stress gradient changes very little in crossing a single grain. In the case of a steep stress gradient it should be more difficult for a crack to get started and to propagate itself. Peterson suggested that a specimen of coarse-grained annealed steel will usually not be reduced in fatigue strength as much percentagewise by the presence of a notch as will a similar specimen of fine-grained heat-treated steel.[6]

Peterson also stated that high-strength, heat-treated steels are highly sensitive to notch effects while normalized steels are less sensitive. This is in direct contradiction to the findings of Dolan and Yen (1). The explanation of this probably lies in the fact that Peterson was not comparing the steels on the basis of equal hardness or tensile strength.

C. W. Beattie (7) found that increasing the *ferrite* grain size of S.A.E. 1020 steel resulted in a lowering of the tensile strength, yield strength, per cent reduction of area, and endurance limit.

Similar tests were performed by H. B. Oehler (8) on S.A.E. 1050 steel. The hardness of all specimens was held to approximately 95 Rockwell B. The endurance limit (unnotched), Charpy impact values, per cent elongation, and reduction of area were all found to increase with a decreasing pearlite grain size. The tensile properties showed one inconsistency in that the largest pearlite grain size had the highest yield strength. The other yield strengths increased as the grain size was decreased. The results of notched and unnotched fatigue tests showed a tendency for the steel to become more notch sensitive (6) as the pearlite grain size decreased. Tensile strengths of the various groups were approximately equal.

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[6] It should be noted that the concept of grain size as used by Peterson was apparently *not* the austenitic grain size but was more closely related to the appearance of the final microstructure.

In a study by R. E. Lorentz, Jr. (9), the influence of grain size on the mechanical properties of an S.A.E. 2345 steel was investigated for both furnace-cooled and quenched and tempered conditions. The furnace-cooled series showed an increase in yield strength, percentage elongation, reduction of area, endurance limit (unnotched), and Charpy impact value as the prior austenitic grain size was decreased from A.S.T.M. No. 1 to No. 7. Similar trends were observed in the hardened and tempered series, but the differences in elongation, reduction of area, and Charpy impact value for grain sizes from 2 to 7 were very slight. The greatest effects of grain size on mechanical properties were observed when the grain size was very large (less than 1 grain per sq. in. at 100×). Data for the furnace-cooled steel indicated an increase in fatigue "notch sensitivity" as the grain size was decreased but no such trend appeared in the *hardened* and *tempered steel.*

P. Schane (46) found that in the annealed, normalized, or quenched and tempered conditions, fine-grained S.A.E. 1040 steel was superior to the coarse-grained steel in ductility and was "vastly superior" in the Charpy impact test at room temperature.

R. A. Rolf (47) presented data for 0.40 per cent carbon steel, of coarse and fine grain, water-quenched from 1525 F. and tempered at 800 F. to 1200 F. by 100-F. intervals. Tensile and yield strengths of the fine-grained steel were 5 to 10 per cent less than for the coarse-grained, but the ductility and toughness were increased. For the 800 F. draw temperature, the fine-grained steel gave Charpy impact values (room temperature) three times as great as those of the coarse-grained steel. Scott (48) reported that a similar effect was found for very hard steels.

E. C. Bain (49) showed that coarse-grained steels gave lower values in the Charpy impact test (room temperature) than fine-grained steels, whether quenched and tempered or austempered. The hardness of the specimens tested was 50 Rockwell C.

J. H. Hollomon, L. D. Jaffe, D. E. McCarthy, and M. R. Norton (2) made a rather extensive examination of S.A.E. 3135 and NE 8735 steels, heat-treated to produce various structures (martensite, martensite plus ferrite and pearlite, and martensite plus bainite) all tempered to the same hardness before testing. These investigators concluded that steels having a tempered martensitic structure had mechanical properties superior to those of other structures. At a given tensile strength the tempered martensites had the highest yield strength, the highest ductility, the highest endurance limit, and the greatest toughness under severe conditions. It should be noted that these conclusions were based primarily upon steels below a hardness of 50 Rockwell C scale and having carbon contents less than 0.50 per cent.

Gensamer, Pearsall, Pellini, and Low (4) discussed the tensile properties of pearlite, bainite and spheroidite. A relation between the strength of steel and the average spacing of the carbide particles was proposed as follows: "The resistance to deformation of a metallic aggregate consisting of a hard phase dispersed in a softer one is proportional to the logarithm of the mean straight path through the continuous phase." By the mean straight path was meant the average distance from one hard particle to another along a straight line taken at random. It was further concluded that the *amount* of the hard, dispersed phase was of less consequence than the mean path through the soft continuous phase.

From this concept of the strength of metallic aggregates one would predict that the prior austenite grain size *alone* has little if any effect on the tensile properties of tempered martensite.

A relatively small number of papers have been surveyed in this Appendix. From even this limited number, it appears evident that there has been a definite lack of agreement on the effects of austenite grain size on the mechanical properties of steel. In many of the early studies there was a tendency to attach undue significance to the results of room temperature Charpy impact tests. The appreciable increase in hardenability usually observed with an increase in the austenite grain size suggests that further study of this old problem could prove profitable.

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DISCUSSION

MR. J. M. HODGE (*presented in written form*).[1]—These studies by Messrs. Sinclair and Dolan will be, I am sure, of great interest to the metallurgical profession, as they are on a subject which is of fundamental importance to metallurgists; the relationship between microstructure and properties. The principal conclusions as to the effect of austenitic grain size on properties in tempered martensitic and in pearlitic steels, namely, that it makes little difference in the tempered martensitic steels and on the other hand that it is tremendously important in pearlitic steels, are, I believe, now well established by these studies.

I believe, however, that it should be pointed out that, in all probability, all of the structures described as tempered martensites are not fully tempered martensite nor are all of the structures described as pearlitic, actually fine pearlite. These departures from the pure structures should, I believe, be taken into consideration in evaluating the relationships between microstructure and properties. The S.A.E. 1045 steel, as mentioned by the authors, contained some fine pearlite as quenched, and the relatively high transition temperature on impact testing as compared with those for the S.A.E. 2340 and 3140 steels is undoubtedly a reflection of the presence of this fine pearlite in the tempered microstructure. In the S.A.E. 1045 steel with ferrite (Series 15), likewise, the authors mention the presence of fine pearlite at the grain boundaries, and the presence of this carbide phase at the grain boundaries might well explain some of the differences shown in their data between the effects of ferrite on the properties of tempered martensite in the S.A.E. 1045 and 2340 steels.

There are, furthermore, distinct and possibly significant differences among the pearlitic microstructures. I believe that most metallurgists would agree that the structure of the oil-quenched S.A.E. 1045 steel is probably largely fine pearlite, but the structures of the air cooled S.A.E. 2340 and 3140 steels, would, I think, generally be referred to as upper bainite.

This therefore permits a comparison of the properties of fine pearlite and upper bainite microstructures at the same hardness level, and it is of particular interest that the upper bainitic structures seem definitely superior, particularly in respect to toughness; at any rate, when tempered at these relatively high tempering temperatures.

MR. PETER R. KOSTING (*presented in written form*).[2]—The importance of the work of Dolan and his associates cannot be over emphasized. The engineer needs such data as these to help in the selection of material for particular applications. The benefits of having a certain microstructure in steel for a particular component must be capable of being evaluated. The engineer is not always privileged to build up personal experience by having components fail because of the lack of such expensive benefits in the steel. Such data as are in this paper should be helpful in this evaluation.

In studying the fatigue data, it is apparent that for any one particular medium-carbon low-alloy heat-treated steel, a processing which improves ductility and toughness, also improves resistance to fatigue, even under a multiaxial stress system. The comparison of steels of different heats or of different alloy composition, however, is not as simply made because of slight differences in strength levels and in microstructures. It would be helpful to make corrections in the data for the slight differences in strength levels in order to simplify intercomparisons. If the test procedures give data representative of fundamental properties of the steel, that is, reasonably unaffected by size effects, these intercomparisons should give useful reliable information. Thus, when series 11 and 13 are compared, these two being selected out of all the steels because of the similarity in microstructure, as revealed in Figs. 20 and 22, the averaged data show a difference only in the region of finite life. In view of the note by Ransom and Mehl[3] based on the results of a statistical study of fatigue, it appears that this difference between series 11 and 13 might be non-existent. It might also be that the differences in slopes of all of the S-N curves for unnotched specimens apparent in Figs. 7, 8, and 9, are not real.

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MESSRS. G. M. SINCLAIR AND T. J. DOLAN (*authors' closure*).—The authors wish to thank Mr. Hodge and Mr. Kosting for their very interesting and helpful discussions.

The first point raised by Mr. Hodge is well taken, namely, that it would be well to emphasize the fact that the microstructures considered in this study were probably not 100 per cent tempered martensite or 100 per cent fine pearlite. The authors used the term tempered martensitic *type* of structure to describe those cases in which practically all carbides were spheroidal in shape. Similarly, when most of the carbides present were platelike in form a general classification as a "pearlitic" type of structure was used, although, as Mr. Hodge suggested, "upper bainite" may have been a more exact metallographic term for the structures obtained in the air cooled S.A.E. 2340 and S.A.E. 3140 steels when the austenitic grain size was large. It is the belief of the authors, however, that the final shape and distribution of the carbide particles in the microstructure are of fundamental importance in determining the mechanical properties of steel and that an analysis of the mechanical behavior based on microstructure might justifiably be simplified by considering whether the carbides are spheroidal, platelike or some mixture thereof. When tempered to the same hardness level, the structures termed upper bainite by Mr. Hodge show a toughness superior to that of fine pearlite, as he pointed out. It seems probable that the rather high tempering temperatures used for the upper bainite would result in a partial spheroidization of the initially fine platelike structure and thus might improve the relative toughness of these steels.

With regard to Mr. Kosting's suggestion, the authors have tried to simplify the intercomparison of the yield strength and the endurance limit data, corrected for small differences in strength level, by listing the *ratios* of yield strength or endurance limit to the tensile strength for each steel (for example, Fig. 14). No attempt was made to correct impact data for these slight differences in tensile strength. Mr. Kosting is correct in his assertion that the slopes of the S-N curves are only approximate since they are based on a relatively small number of specimens.

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EFFECT OF VARIATIONS IN NOTCH ACUITY ON THE BEHAVIOR OF STEEL IN THE CHARPY NOTCHED-BAR TEST*[1]

By N. A. Kahn,[2] E. A. Imbembo,[2] and F. Ginsberg[2]

Synopsis

This paper presents and discusses data on the effects of small variations, in the notch root radius of Charpy V- and keyhole-type specimens on the energy absorption level and location of the transition temperature region of a number of steels. The principal objective was to establish tolerances for the root radius dimension of the V-notch, for which no standards exist at the present time. The materials used in the investigation consisted of Navy medium and high-tensile strength steels and S.A.E. 1045 and 4620 steels, heat treated to tensile strength levels of approximately 60,000, 70,000, 85,000, 110,000, or 155,000 psi. For each steel and condition, energy-temperature relationships were established for V-notch radii of 0.006, 0.0075, 0.009, 0.010, 0.011, 0.012, 0.015, 0.017, and 0.021 in. and for keyhole diameters produced by Nos. 46, 47, and 48 drills. The effect of polishing the base of the A.S.T.M. standard 0.010 in. radius V-notch was also investigated. Equipment and techniques for preparing the notches and conducting the tests are described.

The Problem

The literature on notched-bar testing indicates that Charpy keyhole- and V-notch specimens have been extensively used for evaluating the notch-sensitivity characteristics of metals at normal, subnormal, and elevated temperatures. The form and dimensions of these specimens are given in the Tentative Methods of Impact Testing of Metallic Materials (E 23 – 47 T).[3] These Methods as well as many others include tolerances for most significant dimensions but do not specify any for the radius at the root of the V-notch and the diameter of the hole in the keyhole notch. At the present time, tolerances for the radius at the base of the notch with A.S.T.M. Methods[3] are not specified, in the absence of sufficient data to support the value or significance of such tolerances.

Actually, some investigators have expressed the opinion that small deviations in the notch root radius, particularly that of the V-notch, will influence the energy value, transition temperature, and reproducibility of results. Others have raised the question as to the influence of machine finish at the root of the V-notch on the results obtained.

In conventional methods of cutting the notch, the resulting machining marks or scratches at the root are not in the optimum direction with respect to the breaking stresses, and it has been claimed that variations in the smoothness of the surface will influence the test results. In this connection, one laboratory (**1**)[4] has attempted to eliminate effects of machining marks by polishing the base of the 0.010 in. radius V-notch with a rotating 0.020 in. wire using No. 600 carborundum abrasive. However, another laboratory (**1**) has reported that there appear to be no significant differences between results obtained with unpolished and polished notches.

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[1] The opinions or assertions contained herein are those of the authors and are not to be construed as official or reflecting the views of the Navy Department or the Naval Service at large.

[2] Head, Senior, and Associate Metallurgists, respectively, Material Laboratory, New York Naval Shipyard, Brooklyn, N. Y.

[3] 1949 Book of ASTM Standards, Part 2, p. 1035.

In view of the apparent lack of data on the effect of small variations in notch root radius and the influence of notch finish, the laboratory with which the authors are associated was authorized by the Bureau of Ships, Navy Department, to investigate the effects of these variables on the energy level and transition temperature characteristics of a number of steels ranging in tensile strength from 60,000 to 155,000 psi. The investigation also was sponsored to acquire data which would provide a basis for the incorporation of tolerances for the notch root radius dimension of Charpy specimens in A.S.T.M.[3] and Navy Department specifications.[5]

REVIEW OF PREVIOUS WORK

A review of the literature indicates that there is considerable published data on the Charpy notched-bar test some of which deals specifically with the effects of variations in notch acuity on the energy-absorption and transition-temperature characteristics of steel.

Greaves and Jones (**2**) presented data showing the influence of changes in the notch root radius of the Charpy specimen on the energy-temperature relationships for ingot iron. The variations included root radii of 0.01, 0.04, 0.16, 0.4, and 7.9 in., all of the notches being cut to a depth of 0.079 in. Results of their work showed that the greater the sharpness of the notch, the lower is the curve of total work and the higher is the temperature at which the drop-off in energy occurs.

R. Mailander (**3**) investigated the notch-sensitivity characteristics of carbon and chromium-nickel steels using several sizes of Charpy specimens with radii at the base of the notch of 0.004, 0.01, 0.04, 0.08, 0.12, and 0.2 in. The notches were cut to half the depth of the bar. Results of tests conducted at several selected temperatures indicated that the smaller the size of the notch, the lower is the energy-absorption value. In the case of a 0.12 per cent carbon steel tested at −70 C., the energy values associated with cleavage-type fractures were not appreciably affected by variations in notch radius.

In a subsequent paper, Mailander (**4**) presented Charpy data on low- and medium-carbon steels tested at a number of temperatures using standard-size specimens with notch radii of 0.02, 0.04, and 0.08 in., and notch depths of 0.08, 0.12, and 0.2 in. In general, his results indicated a shift in the energy-temperature curve to a region of lower temperature with an increase in the radius of the notch. The energy values were generally lower for the sharper notches.

Crafts and Egan (**5**) investigated the Charpy V-notch properties of S.A.E. 1020X steel using standard-size specimens with root radii of 0.010 and 0.039 in. and notch depths of 0.079, 0.139, and 0.199 in. Tests were conducted at room temperature, −50 C., and −78 C. using various striking velocities and initial energies. The results indicated that the critical transition range was shifted to higher temperatures by increase of striking velocity, decrease of initial energy, and decrease of notch radius. In general, higher energy values were associated with the larger notch radius.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 647.

[5] General Specifications for Inspection of Material, Appendix II, Metals, Part A—Definitions and Tests, issued by Navy Department, April 1, 1947.

Jackson, Pugacz, and McKenna (6) conducted Charpy tests on carbon and low-alloy steels utilizing standard-size specimens, notched to a depth of 0.079 in. with milling cutters of 0.010 (standard), $\frac{1}{16}$, $\frac{1}{8}$, and $\frac{3}{16}$ in. radii. Their findings indicated that, as the radius of the notch increases, the energy required to fracture the specimen increases.

TABLE I.—CHARACTERISTICS[a] OF STEELS USED IN NOTCH ROOT RADIUS STUDIES.

Code Designation for Material	Type of Open Hearth Steel Selected for Study	Heat Treatment Received[b d]	Yield Strength, 0.1 per cent offset, psi.	Tensile Strength, psi.	Elongation in 1 4 in., per cent	Reduction of Area, per cent
A	Medium strength[g] semi-killed, grade M, U.S. Navy Spec. 46-S-1; June 1, 1945, welding quality	Normalized at 1650 F., stress relieved at 1150 F., furnace cooled.	40 250[c]	63 000	37.9	66.7
B	High tensile strength[h] fully-killed, "vanity type," grade HT, 46-S-1; June 1, 1945.	Normalized at 1700 F., stress-relieved at 1150 F., furnace cooled.	54 000[c]	69 500	42.8	78.0
C		Normalized at 1700 F. hardened[e] at 1600 F., tempered[f] at 1275 F.	77 000	84 000	34.0	80.0
D	Medium carbon (S.A.E. 1045), fully-killed.	Normalized at 1650 F., hardened[e] at 1550 F., tempered[f] at 1175 F.	103 000	112 000	28.0	67.0
E		Normalized at 1650 F., hardened[e] at 1550 F., tempered[f] at 925 F.	135 000	153 000	19.0	60.0
H	Nickel-molybdenum (S.A.E. 4620), fully-killed.	Normalized at 1700 F., hardened[e] at 1600 F., tempered[f] at 1250 F.	102 500	110 500	27.0	71.0
J		Normalized at 1700 F., hardened[e] at 1600 F., tempered[f] at 900 F.	152 500	157 500	18.0	63.7

[a] Tensile properties determined on 0.357-in. diameter specimens machined from $\frac{1}{2}$ by $\frac{1}{2}$-in. bar stock after heat treatment.

[b] Holding time at normalizing and hardening temperature was $\frac{1}{2}$ hr.; at stress relieving and tempering temperatures it was 1 hr.

[c] Values are yield points, psi.

[d] Bar stock was air cooled after normalizing.

[e] Water quenched before tempering.

[f] Air cooled after tempering.

[g] Approximate composition, per cent: C = 0.18; Mn = 0.48.

[h] Approximate compostion, per cent: C = 0.13; Mn = 1.10; Si = 0.31; V = 0.03; Ti = 0.012.

The work of these investigators may be summarized by stating that the smaller the notch root radius, the lower is the energy-absorption value and the higher is the transition temperature corresponding to the change from shear- to cleavage-type fracture. However, Bagsar (7) presented data on the Charpy V-notch properties of mild steel plate which indicate that the transition temperatures and energy values become independent of the notch root radius for radii of 0.01 in. or smaller. He employed standard-size specimens, V-notched to a depth of 0.197 in. (half-depth), with notch radii of 0.0008, 0.0015, 0.003, 0.006, 0.010, 0.020, 0.030, and 0.085 in. Bagsar plotted the log of transition temperatures in deg. R. *versus* the log of notch root radius in inches, and the resulting curves for mild steel show that the transition temperature remains constant for radii less than and including the standard 0.010 in. radius, but, for notch radii greater than 0.010 in., the transition temperature decreases linearly with increase in notch radius. He states that the insensitivity of Charpy tests to changes in notch root radius in cases where it is less than 0.010 in. can be utilized for reducing the scatter in the test results and suggests that this can be accomplished by specifying the notch root radius dimension for V-notched Charpy coupons to be, say, 0.010 +0.000 −0.005, in. instead of specifying 0.010 in. as in present practice.

It is to be noted that the variations in notch root radius studied by the various investigators were relatively large, and the data give little indication, with the exception of Bagsar's work, as to what might be expected when the notch root radius deviates from that of the standard V- and keyhole-notches by small increments of the order of a few thousandths of an inch. While Bagsar's work is significant, it was confined to only one type of steel and was based on a notch depth of 0.197 in. instead of the standard depth of 0.079 in. Hence, the available data are considered inconclusive from the standpoint of providing a basis for the establishment of tolerances for notch root radii in Charpy specimens.

TESTING PROGRAMS

The testing program consisted of determining the Charpy energy-temperature relationships, which define the change from ductile to brittle behavior, of four steels ranging in tensile strength from approximately 60,000 to 155,000 psi., using standard-size specimens in which the following variations in notch acuity or notch finish were incorporated:

1. V-notch root radii (R) of 0.006, 0.0075, 0.009, 0.010 (standard), 0.011, 0.012, 0.015, 0.017, and 0.021 in.
2. Diameters (D) of hole in keyhole-notch as produced by Nos. 46 (0.081 in.), 47 (0.079 in., standard) and 48 (0.076 in.) drills.
3. Polishing the root of the V-notch as applied to the standard radius.
4. Machining the standard radius V-notch in a single cut as compared to multiple cuts.

MATERIALS INVESTIGATED

Four steels in the form of ½ by ½ in., hot-rolled bar stock were procured to provide test material for the investigation, as described in Table I.

All bars of each steel were rolled from the same billet in order to insure greater uniformity from bar to bar and were furnished in approximately 20-ft. lengths.

METHODS OF TEST AND TEST DATA

Selection and Treatment of Material:

A number of bars of each steel were examined for macrostructure and analyzed for chemical composition; the required amount of material for the investigation was selected on the basis of uniformity of these characteristics. For each steel and condition, five bars were used for the preparation of Charpy V-notch specimens and two bars for keyhole-notch specimens. In the case of the medium and S.A.E. 1045 steels, one additional bar was used in obtaining supplementary data on the relative effect of single and multiple cuts in milling the V-notch.

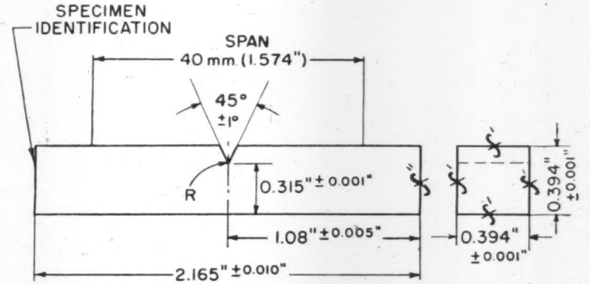
Experimental heat treatment was conducted on each steel for the purpose of establishing suitable heat treating cycles for homogenization or for the development of specific tensile properties. The seven heat treatments evolved and applied to bar coupons from the four steels are also given in Table I, together with the tensile properties obtained by each treatment and the code letters assigned to each heat-treatment modification.

The bars were cut to provide blanks 2¼ in. long for the Charpy specimens. The blanks designated for each notch radius were taken from selected locations in individual bars on the basis of random distribution tables. The blanks from each bar were identified in such a way that the notches were all cut into the same surface of the bar. The coupons were then heat treated in accordance with the procedures indicated in Table I.

Technique for Machining V-notch Specimens:

The V-notch specimens were machined to the A.S.T.M. dimensions and tolerances given in Fig. 1. Also, a tolerance of ±0.001 in. was imposed on the section thickness beneath the notch. In arriving at the 0.394 by 0.394-in. cross-section, an equal amount was removed from each face of the $\frac{1}{2}$ by $\frac{1}{2}$-in. bar stock. The notches were machined with $2\frac{3}{4}$-in. multiple-tooth milling cutters, tipped with Tantung inserts.

For any notch size, five specimen blanks were simultaneously notched by clamping them in a specially designed jig mounted on the milling machine table. As indicated in Fig. 1, the milled end of the specimen was banked against a stop in the jig in order that the notch would always be cut at the same distance from one end, thereby facilitating subsequent alignment of specimens in the impact testing machine. In machining each notch size, two milling cutters were employed—one a roughing and the other a finishing cutter. The notches were (1) cut to a depth of 0.060 in. using the roughing cutter, (2) cut to an additional depth of 0.012 in. using the same cutter, and (3) finally cut (approximately 0.007 in.) with the finishing cutter to bring the section thickness beneath the notch to 0.315 ±0.001 in. For one phase of the investigation, the standard notch (0.010 in. radius) was machined to the required depth in one step in order to determine the relative effect of single and multiple cuts on the energy-temperature relationship.



f' = longitudinally ground finish, f'' = milled end for banking against stop in fixture used to hold specimens for notching and against stop in impact testing machine.

FIG. 1.—Dimensions of V-Notch Charpy Specimens.

To check notch contour and root radius of actual cuts, the various size V-notches were cut[6] in $\frac{1}{16}$-in. thick steel strips which were clamped between supporting blocks. The resulting notches were examined on an optical comparator under a magnification of 100×. In photographs of the notch profiles, it was observed that, in all cases, the included angle of the notch conformed to the specified ±1 deg. tolerance and that the contour and radius at the root of each notch conformed very closely to the theoretically correct profile.

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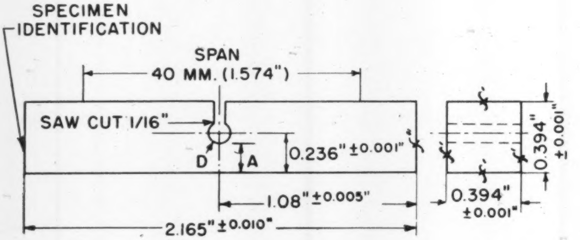
[6] In the opinion of the authors and assisting shop personnel, multiple-tooth cutters were considered more efficient than carbide-tipped fly-cutters, from the standpoint of minimizing chatter and wear, and the results obtained with the multiple-tooth cutters appeared to be superior to those shown by Siemen (8) using carbide tipped fly-cutters

section thickness (*A*) beneath the notch which varies slightly with the hole size.



′ = longitudinally ground finish, *f″* = milled end for banking against stop in drill jig and in impact machine.

FIG. 2.—Dimensions of Keyhole-Notch Charpy Specimens.

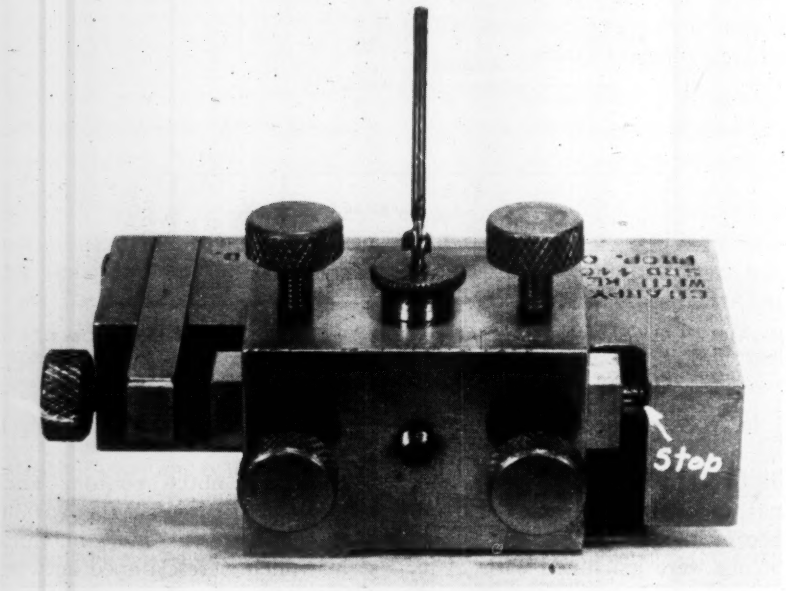


FIG. 3.—Jig Used for Drilling Hole in Keyhole-Notch of Charpy Specimen (Full Size).

Technique for Machining Keyhole-notch Specimens:

The keyhole-notch specimens were machined to the dimensions given in Fig. 2; it is to be noted that the axes of the Nos. 46 and 48 drill holes (*D*) are located the same as that of the A.S.T.M. standard No. 47 drill hole resulting in a

The deviations in hole size from that of the standard were intended to simulate discrepancies which might result from poor machining of the standard notch. The specimens were notched by first carefully drilling the hole and then cutting the slot with a $\frac{1}{16}$-in. milling saw. Holes were drilled to size directly without directly resorting to the use of a smaller drill and then finishing to correct size.

The jig used for drilling the holes is shown in Fig. 3. With such a device, any tendency for the drill to drift is minimized, thereby providing for a uniform thickness to fracture. The introduction of an end stop insures that all specimens are drilled at the same longitudinal distance from one end, which ultimately serves as an aid in accurate alignment of specimens in the impact testing machine. The jig is so constructed that it accommodates replaceable bushings for the different drill sizes.

Circles corresponding to theoretical hole diameters were superimposed on photographs of actual contours of the drilled holes to check for conformity.

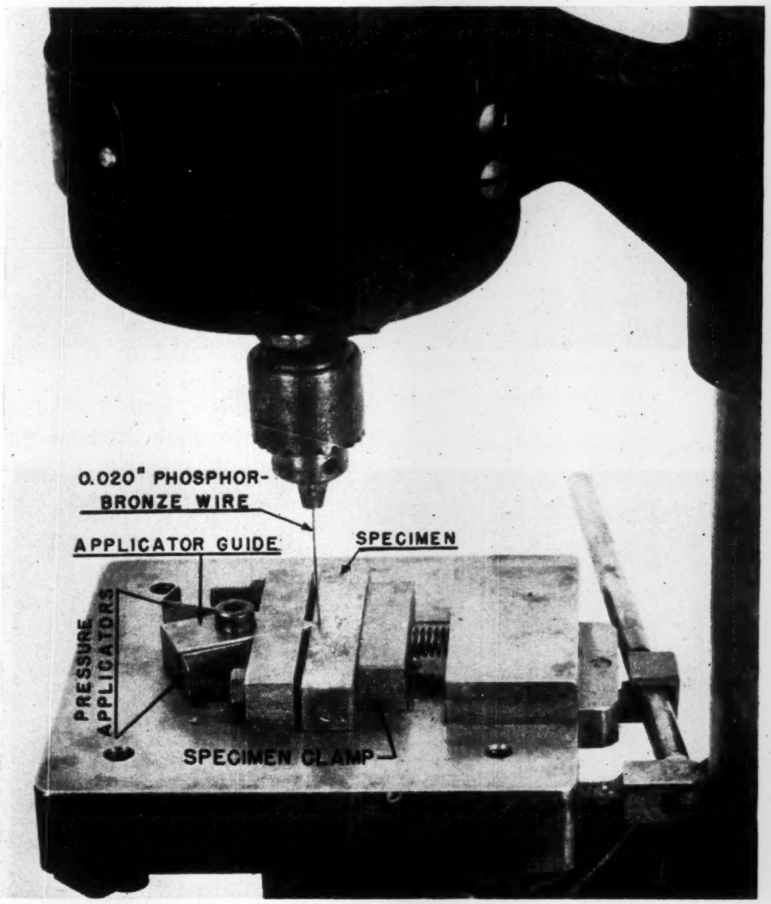
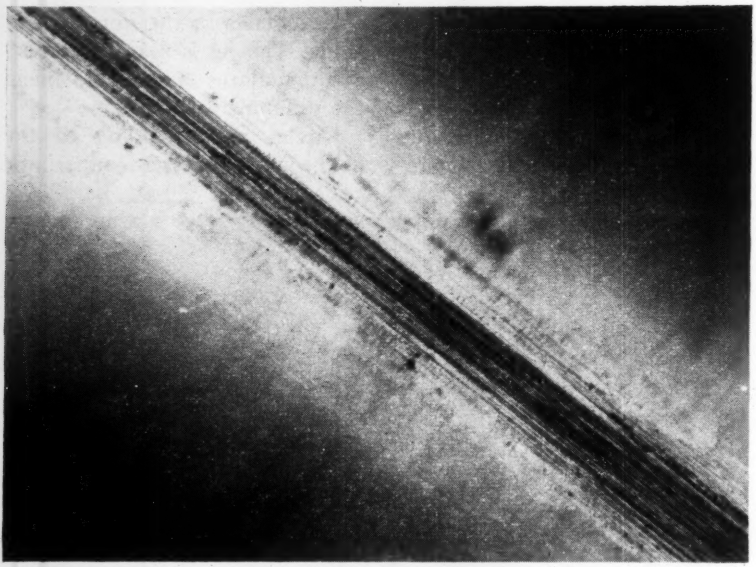


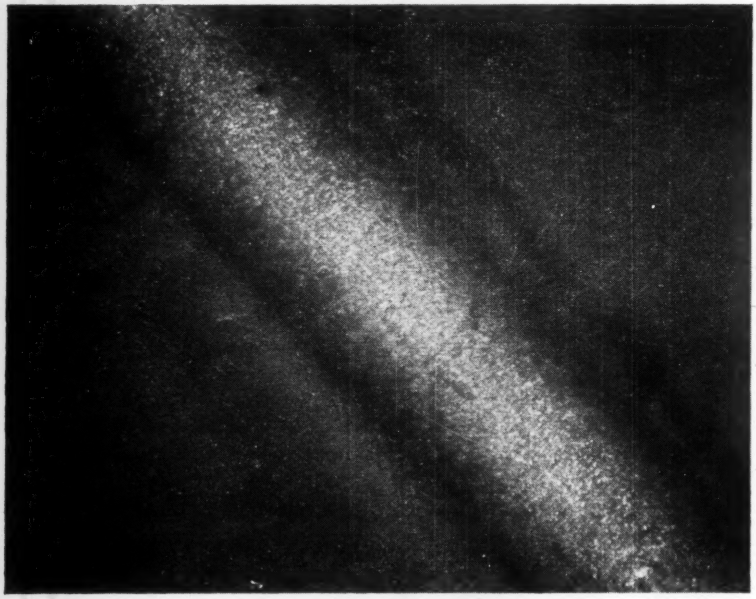
FIG. 4.—Equipment Used for Polishing Root Radius of Standard V-Notch.

Inspection Procedure:

During the course of notching, the specimens were periodically inspected on



As-Machined ×50 (Polarized Light)



After Polishing ×50 (Dark Field)

FIG. 5.—Root of Charpy V-Notch in Machined and Polished Conditions.

the optical comparator in order to detect any undue wear of the cutters or drills. As a final step in the inspection procedure, all specimens were checked for notch conformity and section thickness beneath the notch.

Technique for Polishing Root of V-notch:

To investigate the effect of polishing the root of the standard 0.010-in. radius V-notch, a $\frac{1}{30}$-hp. precision drill press was adapted in such a manner that the root of the notch could be lapped by means of a rotating 0.020 in. diameter phosphor-bronze spring wire with a suspension of No. 600 carborundum in kerosine as the polishing medium. Figure 4 shows a close-up view of the polishing equipment. The function of the applicator guide is to keep the phosphor-bronze wire confined to the root of the notch. The wire was swabbed with the polishing medium and, with the wire rotating, the pressure applicators were simultaneously pressed in by hand for about 30 sec., during which the specimen was moved up and down by raising and lowering the table. The polishing procedure was completed by rotating the specimen 180 deg. and repeating the above operations. After polishing two specimens, the phosphor-bronze wire was renewed because of some wear and in order to minimize the possibility of altering the root radius size of specimens subsequently polished.

Photomicrographs showing the appearance of the base of the notch in the "as-machined" and polished conditions are presented in Fig. 5. It will be observed that the directional milling cutter marks shown in the "as-machined" specimen have been obliterated by polishing, thereby producing a smooth finish at the base of the notch. Using an optical comparator, under a magnification of 100×, it was established that the polishing procedure did not alter the contour or size of the notch.

Test Equipment and Procedure:

In conditioning the Charpy specimens for tests at various temperatures, several pieces of equipment were employed depending upon the temperature range. For the range of +205 to −100 F., the specimens were conditioned in a liquid bath contained in a sheet metal copper tank, 12 by 6 in. in cross-section and 7 in. deep, with 3 in. of insulation on all sides, and with a removable top cover. The specimens were supported horizontally at the ends on a rack immersed in the liquid. From room temperature to +160 F., a water bath was used; for temperatures above +160 F., glycerol was employed and the bath was heated by an electric immersion heater. For temperatures from room to −100 F., the bath consisted of acetone refrigerated by the direct addition of dry ice. For tests down to −60 F., thermometers were used for temperature measurement; for lower temperatures, a copper-constantan thermocouple and potentiometer were employed, the thermocouple bead being soldered into the notch of a dummy Charpy specimen. Specimens were held at temperature for approximately 20 min. and then transferred to the impact testing machine using precooled tongs.

For test temperatures ranging from −100 to −250 F., it was necessary to construct special equipment, the design of which was based on similar apparatus developed by the Esso Engineering Department of the Standard Oil Development Co., Linden, N.J. However, a number of changes in the design and mechanical arrangement of the component parts were made in order to improve efficiency and operation. The basic method consists of boiling liquid nitrogen in a coiled tube immersed in a low-freezing-point liquid. The Charpy specimens are placed in the latter, the temperature of which is brought to some

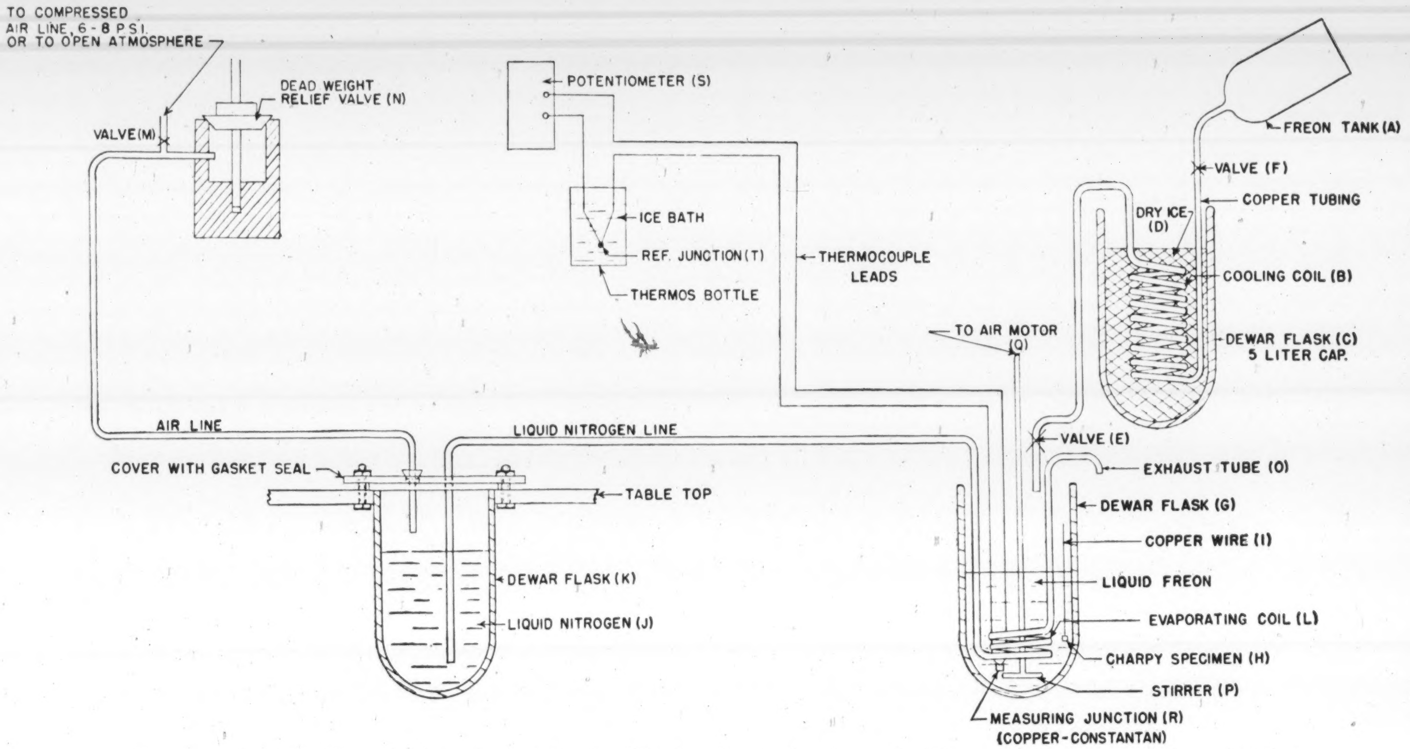

TO COMPRESSED
AIR LINE, 6 - 8 P.S.I.
OR TO OPEN ATMOSPHERE
VALVE (M)
DEAD WEIGHT
RELIEF VALVE (N)
POTENTIOMETER (S)
ICE BATH
REF. JUNCTION (T)
THERMOS BOTTLE
THERMOCOUPLE
LEADS
FREON TANK (A)
VALVE (F)
COPPER TUBING
DRY ICE
(D)
COOLING COIL (B)
DEWAR FLASK (C)
5 LITER CAP.
TO AIR MOTOR
(Q)
AIR LINE
LIQUID NITROGEN LINE
VALVE (E)
EXHAUST TUBE (O)
COVER WITH GASKET SEAL
TABLE TOP
DEWAR FLASK (G)
COPPER WIRE (I)
LIQUID FREON
DEWAR FLASK (K)
LIQUID NITROGEN (J)
EVAPORATING COIL (L)
CHARPY SPECIMEN (H)
STIRRER (P)
MEASURING JUNCTION (R)
(COPPER - CONSTANTAN)

FIG. 6.—Schematic Diagram of Equipment for Conditioning Charpy Specimens for Tests at Temperatures Below −100 F.

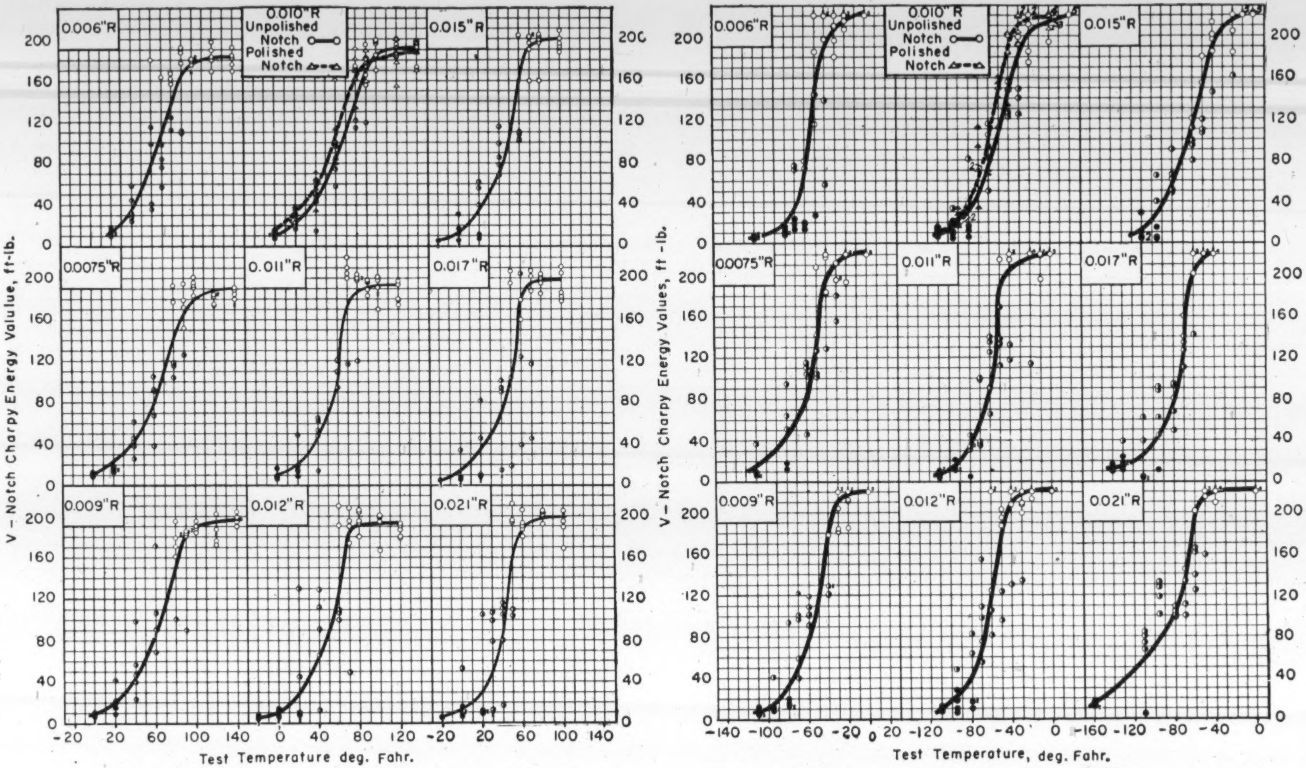
desired value by controlling the amount of liquid nitrogen boiled in the tube. For the liquid bath, the Standard Oil Development Co. utilizes ether for a temperature range of −76 to −175 F. and liquid propane for temperatures of −175 to −319 F. In view of the hazards involved in handling ether and propane in open flasks, the authors substituted liquid Freon-12 (dichlorodifluoromethane) for these media with very satisfactory results. However, the lowest test temperature is limited by its freezing point of approximately −255 F.

A schematic sketch of the equipment is given in Fig. 6. A 50-lb. tank of liquid Freon-12, *A*, supported with its mouth in a lowered position, is coupled to a copper cooling coil *B*, contained in a stainless steel Dewar flask, *C*. The latter is packed with granulated dry ice, *D*, to completely surround coil *B*. With valve *E* closed, valve *F* is opened to permit liquid Freon to flow into coil *B*. The purpose of this arrangement is to cool the liquid Freon to a temperature below its boiling point at atmospheric pressure (approximately −20 F.) so that, when valve *E* is subsequently opened, the Freon entering Dewar flask *G* will be in a liquid state. The Charpy specimens, *H*, are suspended in the Freon bath by fine copper wires, *I*. To bring the Freon bath to the desired test temperature, liquid nitrogen *J* in Dewar flask *K* is pumped into evaporating coil *L* by means of light air pressure controlled by valve *M*. Except for high rates of flow, it is not necessary to use external air pressure since, by throttling valve *M*, the pressure of the gaseous nitrogen in flask *K* can be increased sufficiently to force the liquid to the evaporating coil. To minimize the danger of developing high pressures within flask *K*, a dead-weight relief valve *N* is provided. The nitrogen boiled in coil *L* is exhausted to the atmosphere through tube *O*. By adjusting valve *M*, which controls the flow of liquid nitrogen fed to the evaporating coil *L*, the temperature of the Freon bath may be lowered and maintained at the desired value. The Freon bath is circulated by means of stirrer *P* operated by air motor *Q*. For temperature measurement, the junction of a copper-constantan thermocouple *R* is soldered into the notch of a dummy Charpy specimen. The thermocouple potential is measured by a potentiometer, *S*, using a reference junction, *T*, maintained at 32 F. After holding the Charpy specimens at temperature for approximately 10 min., a specimen was lifted to just above the Freon bath by means of the copper wire, grasped by pre-cooled tongs and then quickly transferred to the impact testing machine.

The operation of transferring and fracturing a specimen did not require more than 2 sec. It is believed that the temperature change, if any, during this interval would be negligible, and the temperature variation between specimens tested at the same temperature would also be negligible. Specimens were fractured in a 220 ft-lb. Riehle pendulum-type impact testing machine under a striking velocity of 18.1 ft. per sec. corresponding to the maximum available kinetic energy of the pendulum. The construction of this machine conforms to A.S.T.M. Methods.[3] In order to align the notch quickly and accurately with respect to the striking edge of the pendulum, the end of the specimen used as the reference point in cutting the notch (Figs. 1 and 2) was banked against a beveled stop on one of the anvil supports. Quintuplicate specimens were tested at each of a number of temperatures so selected as to define the energy-temperature relationships.

Experimental Results:

1. The energy-temperature curves of

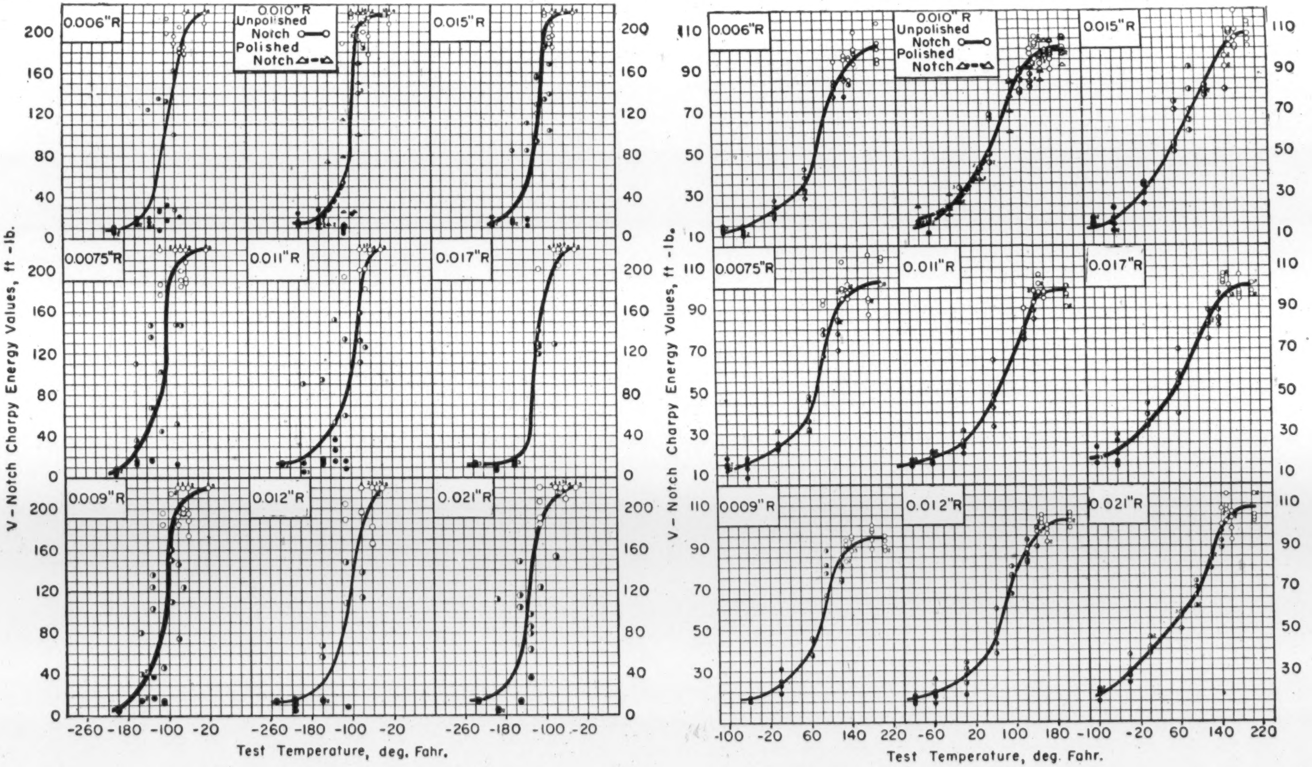


(*a*) Steel A, medium steel, normalized and stress relieved.

(*b*) Steel B, high tensile steel, normalized and stress relieved.

Explanation of Symbols

Unpolished Notch	Polished Notch (0.010"R)
○ 100% Shear Fracture	△ 100% Shear Fracture
◐ Shear-Cleavage Fracture	◭ Shear-Cleavage Fracture
● 100% Cleavage Fracture	▲ 100% Cleavage Fracture



(*c*) Steel C, high tensile steel, water-quenched and tempered to a yield strength of 70,000 to 80,000 psi.

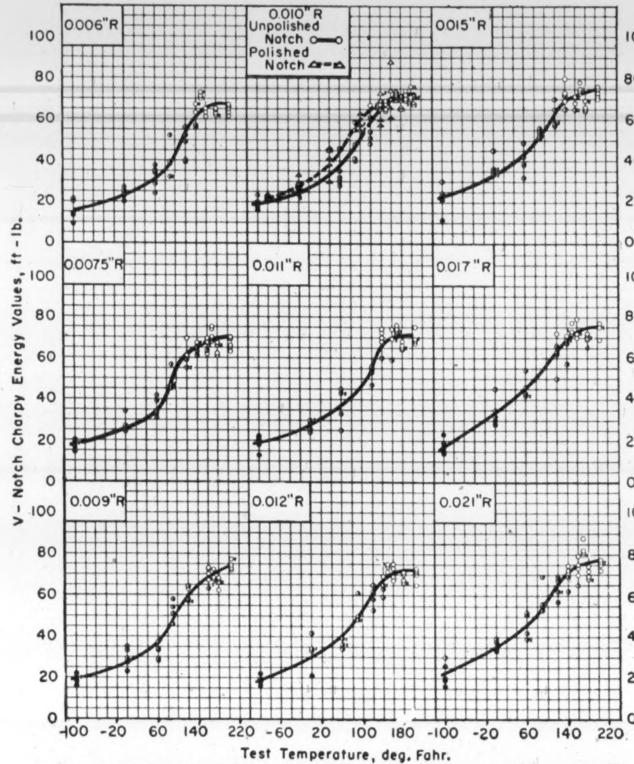
(*d*) Steel D, S.A.E. 1045, water-quenched and tempered to a tensile strength of 105,000 to 115,000 psi.

FIG. 7.—Typical Curves from Original Data Showing Effects of Variation in V-Notch Root Radius on Energy-Temperature Relationship.

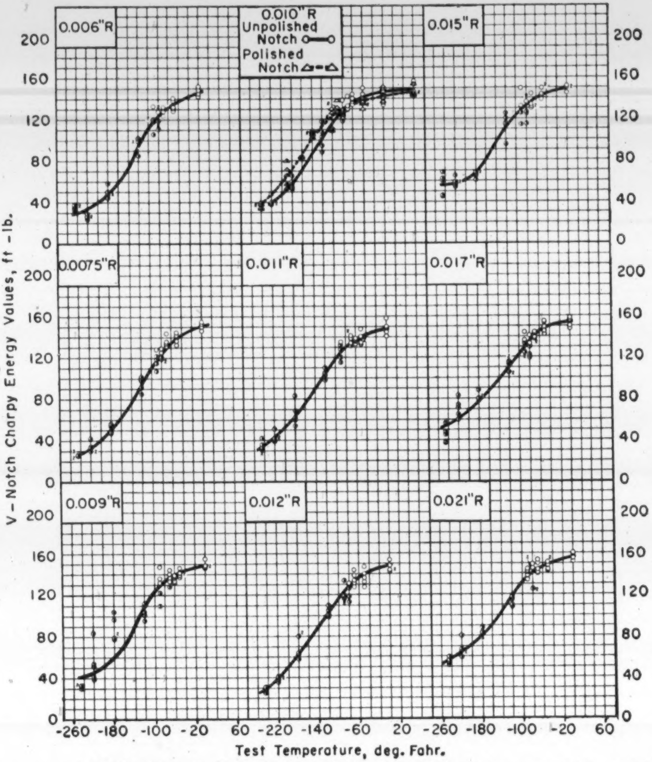
Explanation of Symbols

Unpolished Notch	Polished Notch (0.010"R)
○ 100% Shear Fracture	△ 100% Shear Fracture
◐ Shear-Cleavage Fracture	▲ Shear - Cleavage Fracture
● 100% Cleavage Fracture	▲ 100% Cleavage Fracture

[Continued on p. 632 and 633]



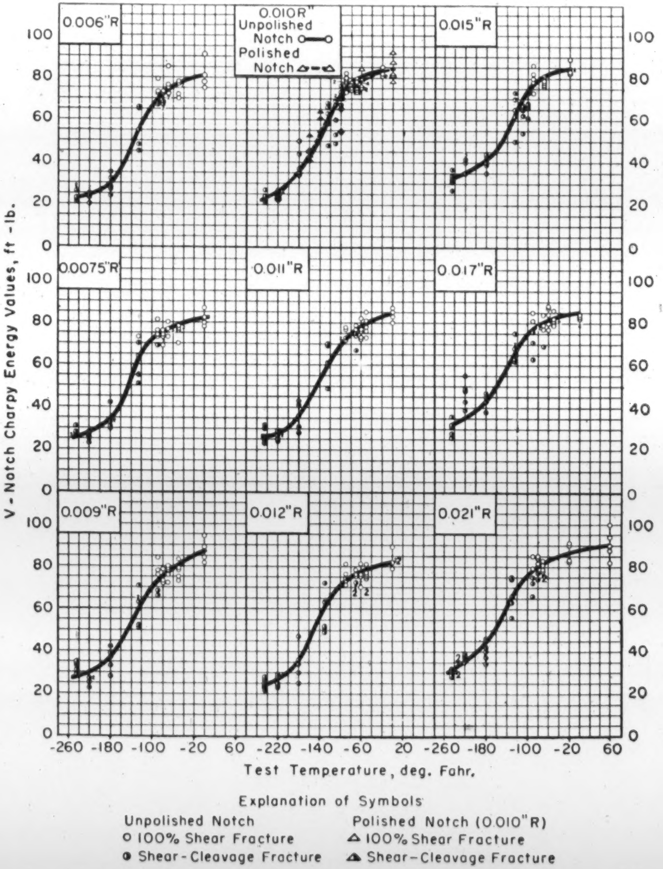
(*e*) Steel E, S.A.E. 1045, water-quenched and tempered to a tensile strength of 150,000 to 160,000 psi.



(*f*) Steel H, S.A.E. 4620, water-quenched and tempered to tensile strength of 105,000 to 115,000 psi.

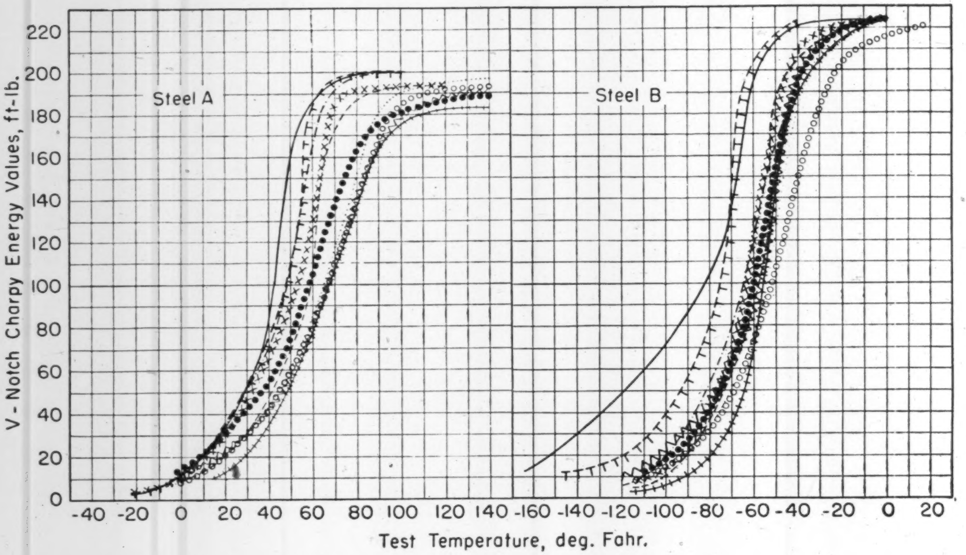
Explanation of Symbols

Unpolished Notch	Polished Notch (0.010" R)
○ 100% Shear Fracture	△ 100% Shear Fracture
◐ Shear-Cleavage Fracture	▲ Shear-Cleavage Fracture
● 100% Cleavage Fracture	▲ 100% Cleavage Fracture



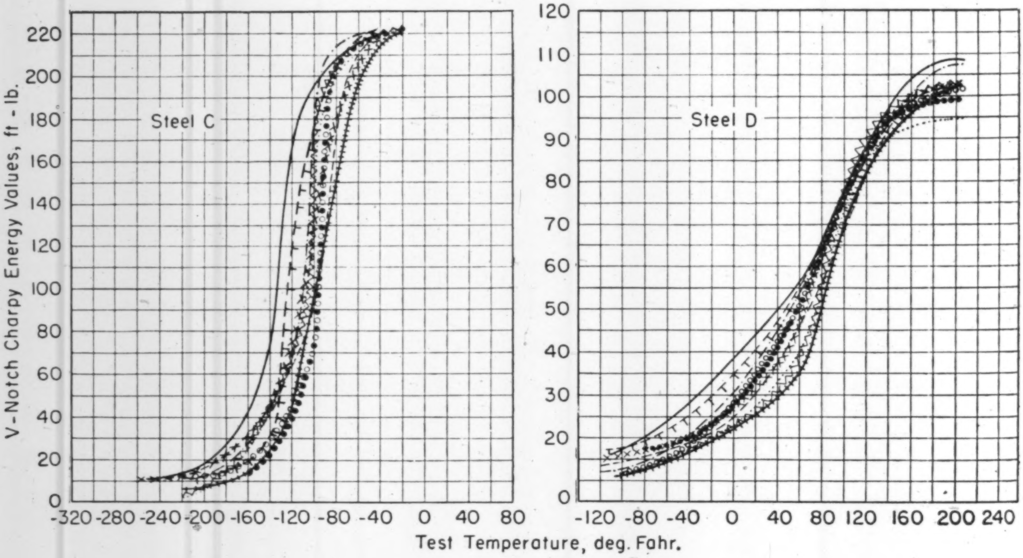
(g) Steel J, S.A.E. 4620, water-quenched and tempered to tensile strength of 150,000 to 160,000 psi.

FIG. 7.—(*Continued*).



(*a*) Steel A, medium steel, normalized and stress relieved.

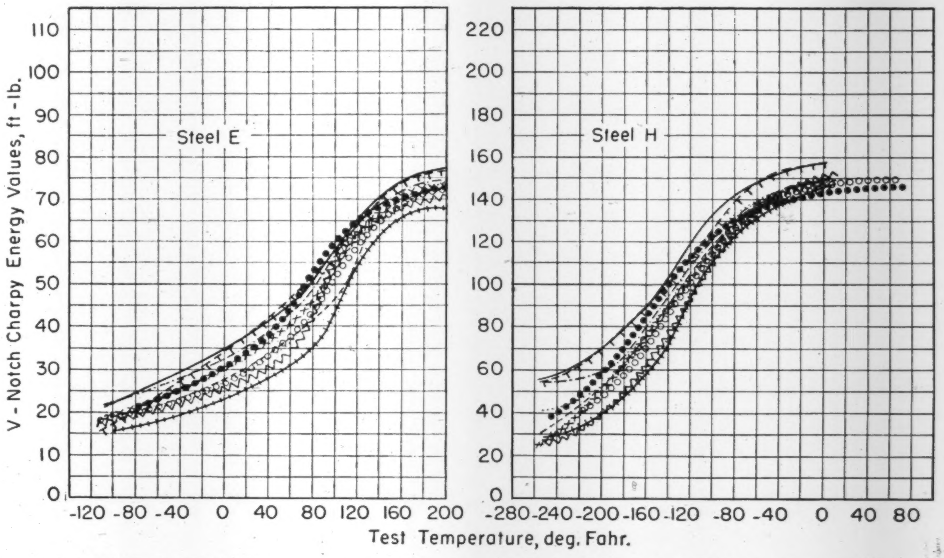
(*b*) Steel B, high-tensile strength steel, normalized and stress-relieved.



(*c*) Steel C, high-tensile strength steel, water-quenched and tempered to a yield strength of 70,000 to 80,000 psi.

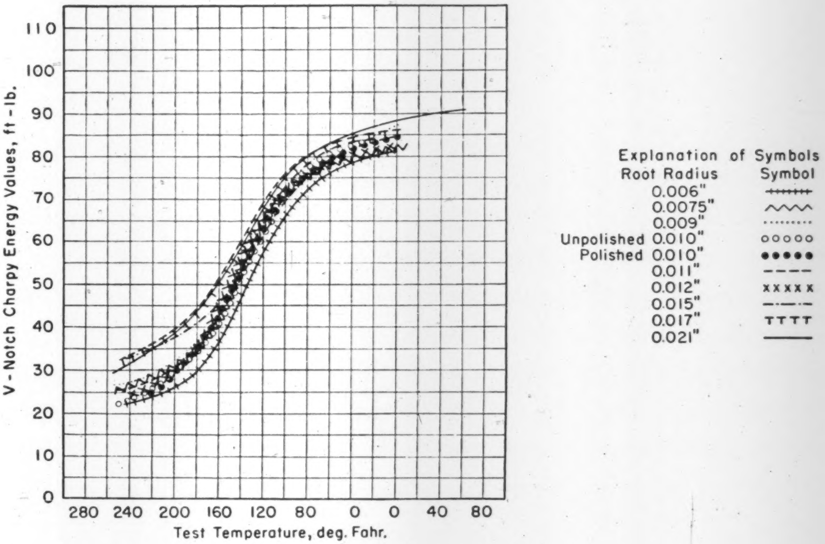
(*d*) Steel D, S.A.E. 1045, water-quenched and tempered to a tensile strength of 105,000 to 115,000 psi.

FIG. 8.—Summary Graphs Showing Effect of Variations in V-Notch Root



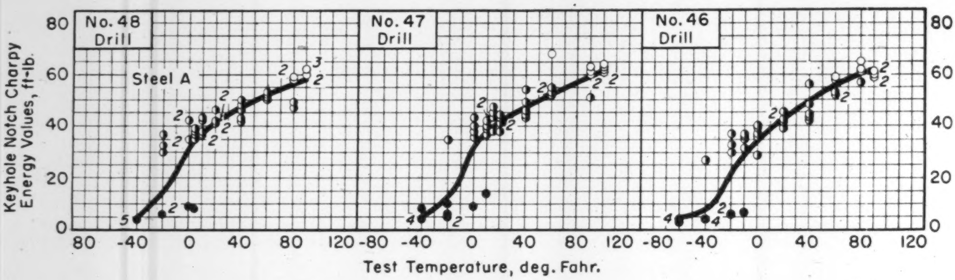
(*e*) Steel E, S.A.E. 1045, water-quenched and tempered to a tensile strength of 150,000 to 160,000 psi.

(*f*) Steel H, S.A.E. 4620, water-quenched and tempered to a tensile strength of 105,000 to 115,000 psi.

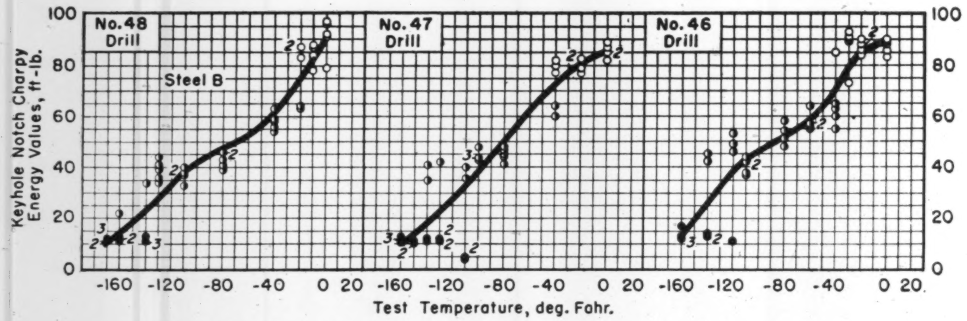


(*g*) Steel J, S.A.E. 4620, water-quenched and tempered to a tensile strength of 150,000 to 160,000 psi.

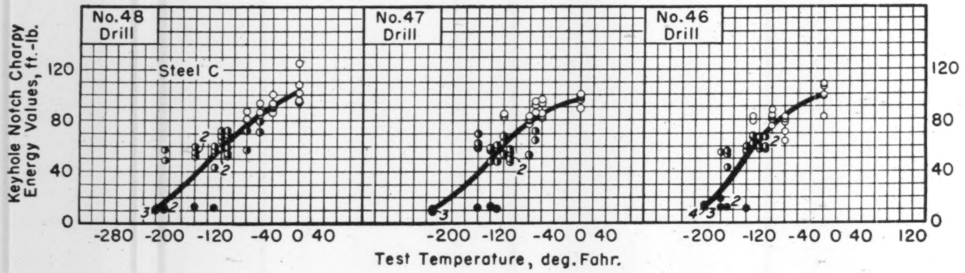
Radius, of Charpy Specimens, on Energy-Temperature Relationships for Seven Steels.



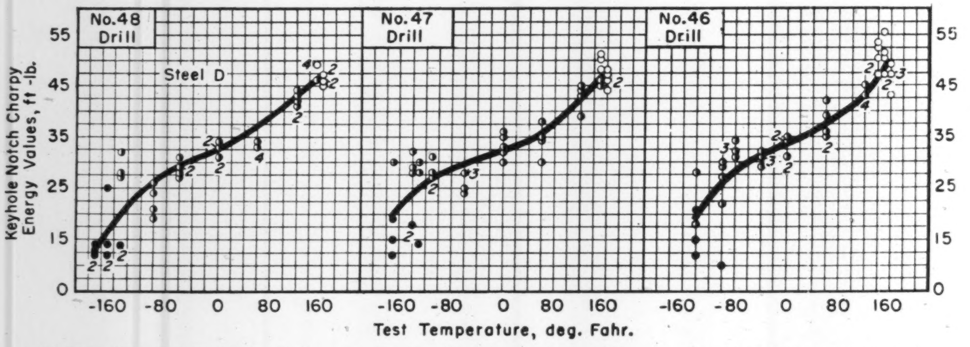
(*a*) Steel A, medium steel, normalized and stress relieved.



(*b*) Steel B, high tensile strength steel, normalized and stress relieved.

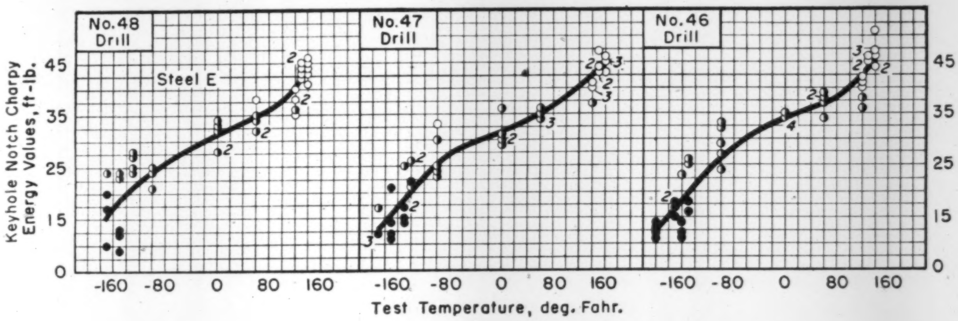


(*c*) Steel C, high tensile strength steel, water-quenched and tempered to a yield strength of 70,000 to 80,000 psi.

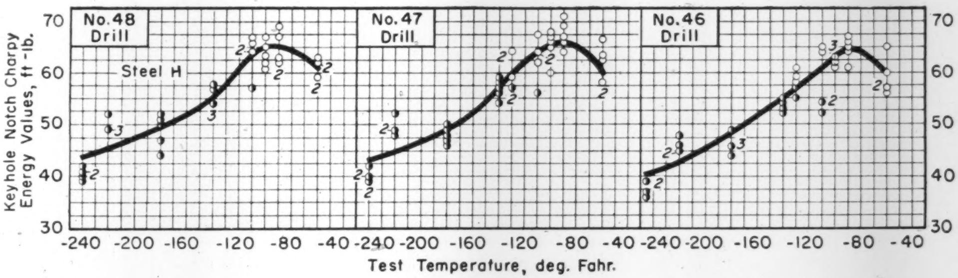


(*d*) Steel D, S.A.E. 1045, water-quenched and tempered to tensile strength of 105,000 to 115,000 psi.

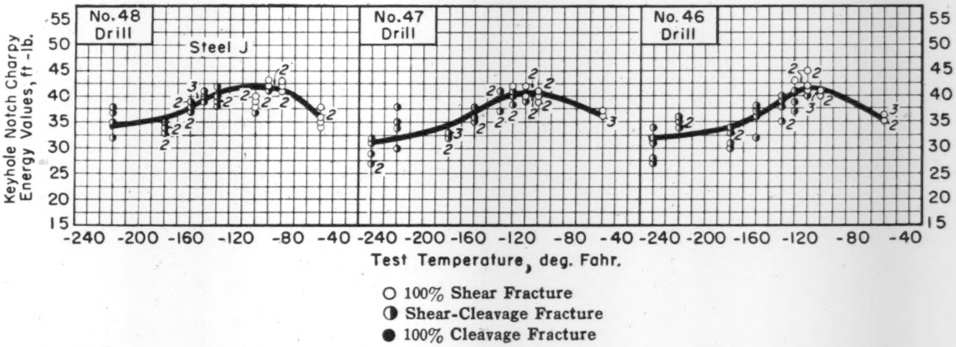
FIG. 9.—Effect of Variations in Drill Hole Size of Charpy



(*e*) Steel E, S.A.E. 1045, water-quenched and tempered to a tensile strength of 150,000 to 160,000 psi.

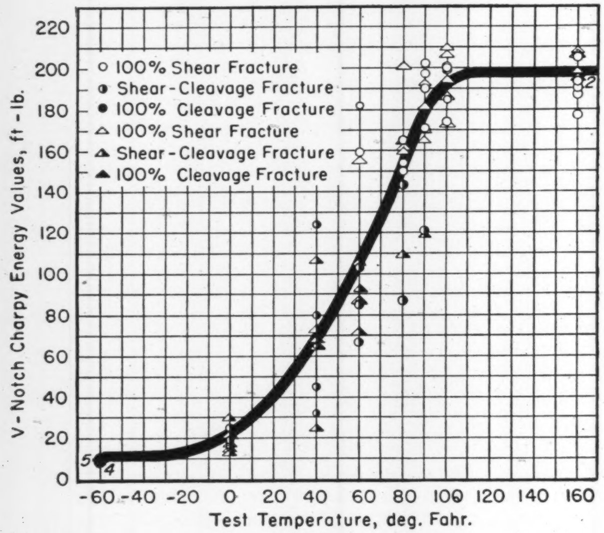


(*f*) Steel H, S.A.E. 4620, water-quenched and tempered to a tensile strength of 105,000 to 115,000 psi.

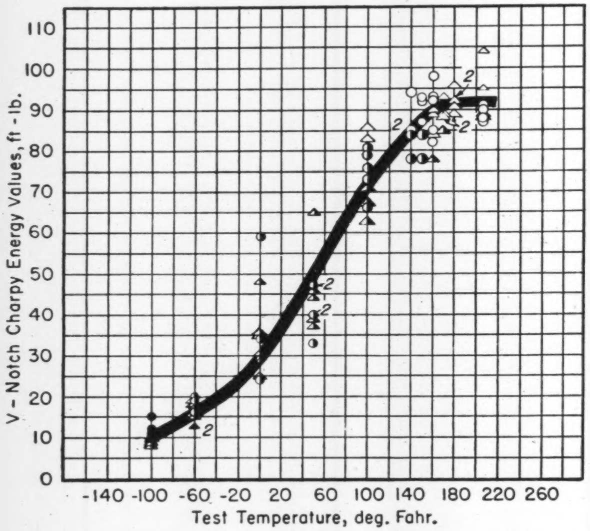


(*g*) Steel J, S.A.E. 4620, water-quenched and tempered to a tensile strength of 150,000 to 160,000 psi.

Keyhole Specimens on Energy-Temperature Relationships.

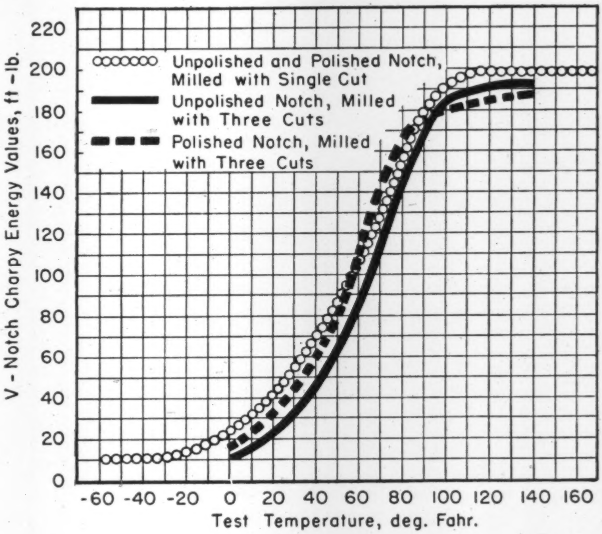


(*a*) Steel A, medium steel, normalized and stress relieved.

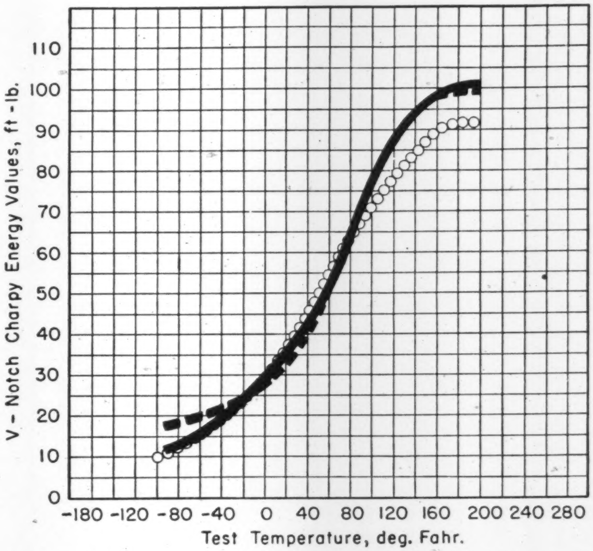


(*b*) Steel D, S.A.E. 1045, water-quenched and tempered to a tensile strength of 105,000 to 115,000 psi.

FIG. 10.—Energy-Temperature Relationship Derived from Charpy Tests on Standard 0.010-in. Radius V-Notch Specimens Milled with One Cut to Required Depth; Unpolished and Polished Conditions.



(*a*) Steel A, medium steel, normalized and stress relieved.



(*b*) Steel D, S.A.E. 1045, water-quenched and tempered to a tensile strength of 105,000 to 115,000 psi.

FIG. 11.—Comparison of Energy-Temperature Relationships for Standard V-Notch Specimen Milled with Single and Triple Cuts in Unpolished and Polished Conditions.

NOTE.—The two curves drawn through average values of results for the unpolished and polished conditions were so close together that they were merged into one.

each steel representing the nine variations in V-notch root radius and the polished 0.010-in. radius notch are presented in Fig. 7.

2. A graphical summary of the curves of Fig. 7 is presented in Fig. 8.

3. Plotted curves showing energy-absorption values obtained at various test temperatures with the three keyhole-notch diameters are given in Fig. 9.

4. Energy-temperature curves derived from Charpy tests of steels A and D using standard 0.010-in. radius V-notch specimens in which the notch was cut to the required depth in one step and tested "as-machined" and also after root polishing are presented in Fig. 10.

5. Figure 11 presents a comparison of energy-temperature relationships for steels A and D using standard 0.010-in. radius V-notch specimens in which the notches were milled with single and triple cuts, and tested in the unpolished conditions.

NOTE.—In recording the original data, some of the V-notch energy absorption values were indicated to be greater than 220 ft-lb. since the corresponding specimens absorbed the maximum available energy of the pendulum without fracturing completely or passing through the anvil supports. This situation occurred only with the high-tensile strength steel tested in the normalized and quenched-and-tempered conditions (Codes B and C, respectively). In these instances, the plotted points in Fig. 7 are marked with upward-pointing arrows at the 220 ft-lb. level.

DISCUSSION OF DATA

In the following paragraphs, the data will be discussed from the standpoint of the effect of variations in notch acuity and notch finish on the transition temperature and energy-absorption value.

Effect of Variations in V-Notch Acuity on Transition Temperature:

From the average energy-temperature curve (see Fig. 7), the temperature corresponding to half of the maximum energy value was taken as the V-notch transition temperature. In the case of steels B and C, the half-energy value was selected arbitrarily at 110 ft-lb. for all V-notches, since the upper level of the curves was not completely defined due to insufficient capacity of the impact testing machine.

The transition temperatures, as defined above, have been plotted against the log of the V-notch root radius in the upper portion of Fig. 12. For steels A, B, and C, it is to be observed that the transition temperature was virtually unaffected by variations in notch root radius ranging from 0.006 to approximately 0.011 in., and that for root radii greater than 0.011 in., the transition temperature decreased with increase in the notch root radius. The results obrained on steels A, B, and C confirm the findings of Bagsar (7) on mild steel. While these steels appear to be insensitive to a reduction in the root radius below approximately 0.011 in., the behavior of steels D, E, H, and J did not follow this pattern. In these steels, the transition temperature decreased continuously with increase in notch root radius from 0.006 to 0.021 in. It may be that the threshold of insensitivity for steels D, E, H, and J occurs at a root radius less than 0.006 in. Bagsar's suggestion, that the insensitivity of Charpy test results due to a reduction in notch root radius below 0.010 in. can be utilized for reducing the scatter in the results by specifying the root radius dimension to be 0.010. +0.000, −0.005 in. would not, therefore, appear to be universally applicable to all steels. In this connection, it is interesting to note that steels A, B, and C (medium and high-tensile strength) represent material of lower tensile strength than steels D, E, H, and J.

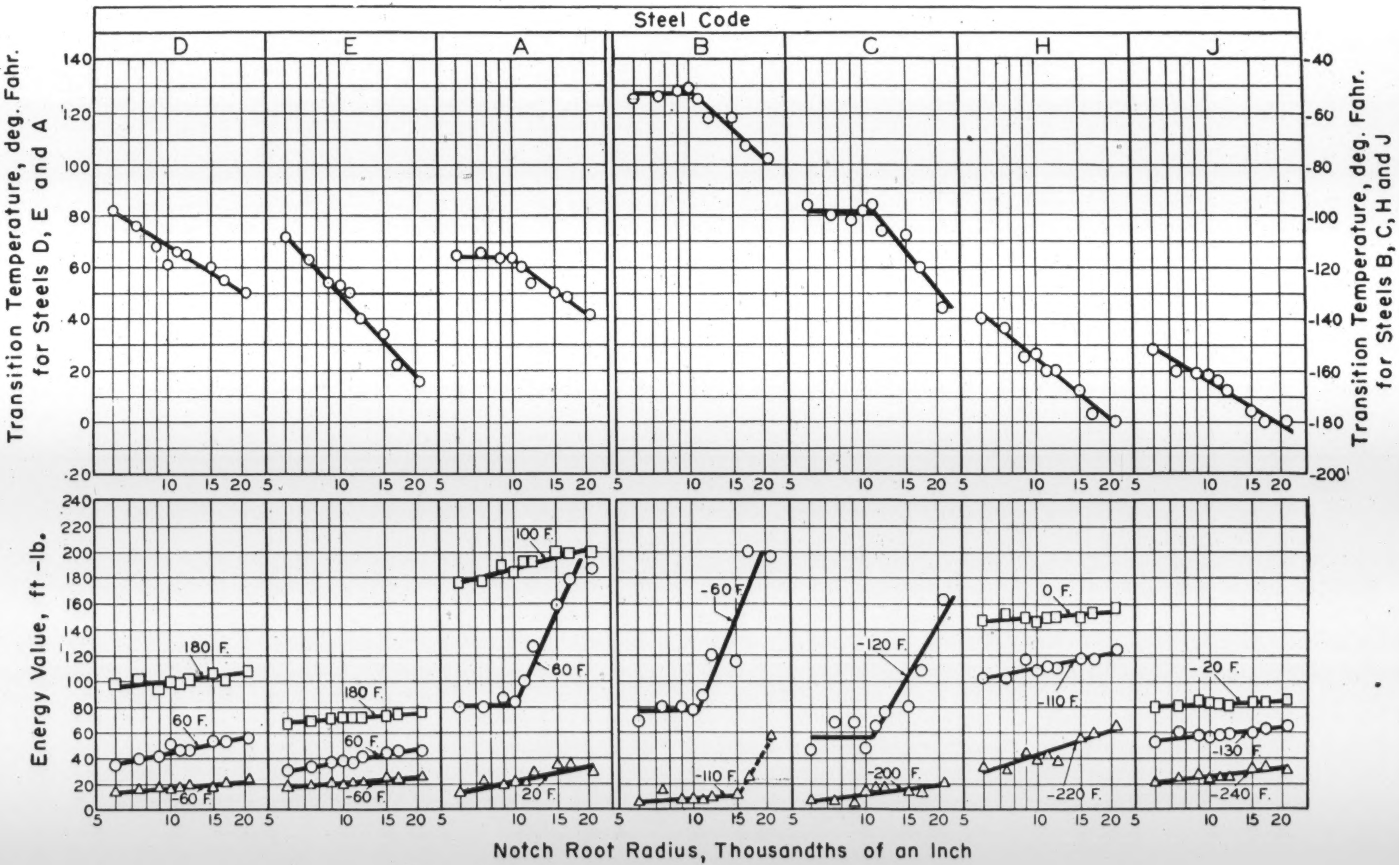


FIG. 12.—Effect of V-Notch Root Radius on Transition Temperature and Energy Absorption Values.

Effect of Variations in V-Notch Acuity on Energy-Absorption:

From the group of energy-temperature curves representing each steel and embracing variations in V-notch root radius of 0.006 to 0.021 in., three temperatures corresponding to high, intermediate, and low energy levels were selected. Energy values at each selected temperature were then taken from the average energy-temperature curves. The values so obtained have been plotted against the log of the V-notch root radius in the lower portion of Fig. 12. In the case of steels B and C, no energy values are shown for a "high" temperature since, as previously indicated, the upper level of the curves for these steels was not adequately defined (see Note on p. 640). Considering the "high" and "low" temperatures corresponding to regions above and below the transition zone, respectively, it is to be observed that the energy-absorption value shows, in general, a continuous but small increase with increase in the notch root radius. For steel B, the energy value for the "low" temperature shows an abrupt increase for root radii greater than 0.015 in. Examination of the individual energy-temperature curves for this steel indicates that the abrupt increase is due to the fact that the selected "low" temperature of −110 F. falls within the transition or scatter region of the energy-temperature curves for the 0.017- and 0.021-in. notches. In regard to the "intermediate" temperature which falls within the transition region (Fig. 12), it is to be observed that, for steels D, E, H, and J, the energy value shows a continuous but relatively small increase with increase in notch root radius, which is in line with the corresponding decrease in transition temperature. In the case of steels A, B, and C, the energy value is at first relatively constant for an increase in notch root radius up to approximately 0.011 in. and then increases with further increase in the root radius. The relatively sharp increase in the energy value for notch root radii greater than approximately 0.011 in., as compared to other steels, is due to the greater steepness of the energy-temperature curves of the former steels as compared to those of the latter. It is to be noted particularly that the energy change with increase in notch root radius at the selected "intermediate" temperatures for steels A, B, and C closely parallels the corresponding change in the transition temperature.

Effect of Variations in Keyhole Diameter on Transition Temperature:

Defining the transition temperature on the basis of the value corresponding to half of the maximum energy level was not applied to the keyhole energy-temperature curves since several did not extend to sufficiently low energy levels. Accordingly, the transition temperature for all keyhole notch sizes of the same steel was taken at an energy level on the average energy-temperature curve approximately midway between the maximum and minimum energy values.

The transition temperatures, as defined above, have been plotted against the keyhole diameter in the upper portion of Fig. 13. With a few exceptions, which may be attributed to experimental variations, it is to be observed that an increase in the hole diameter resulted in a decrease in the transition temperature. A change, either way, of one drill size number from that of the No. 47 did not greatly affect the transition temperature of steels A, B, C, H, and J, the maximum deviation being 10 F. While a reduction in the hole size (No. 47 to 48) likewise had no great effect on the transition temperatures of steels D and E, an increase from No. 47 to 46 resulted in a considerable decrease in the transition

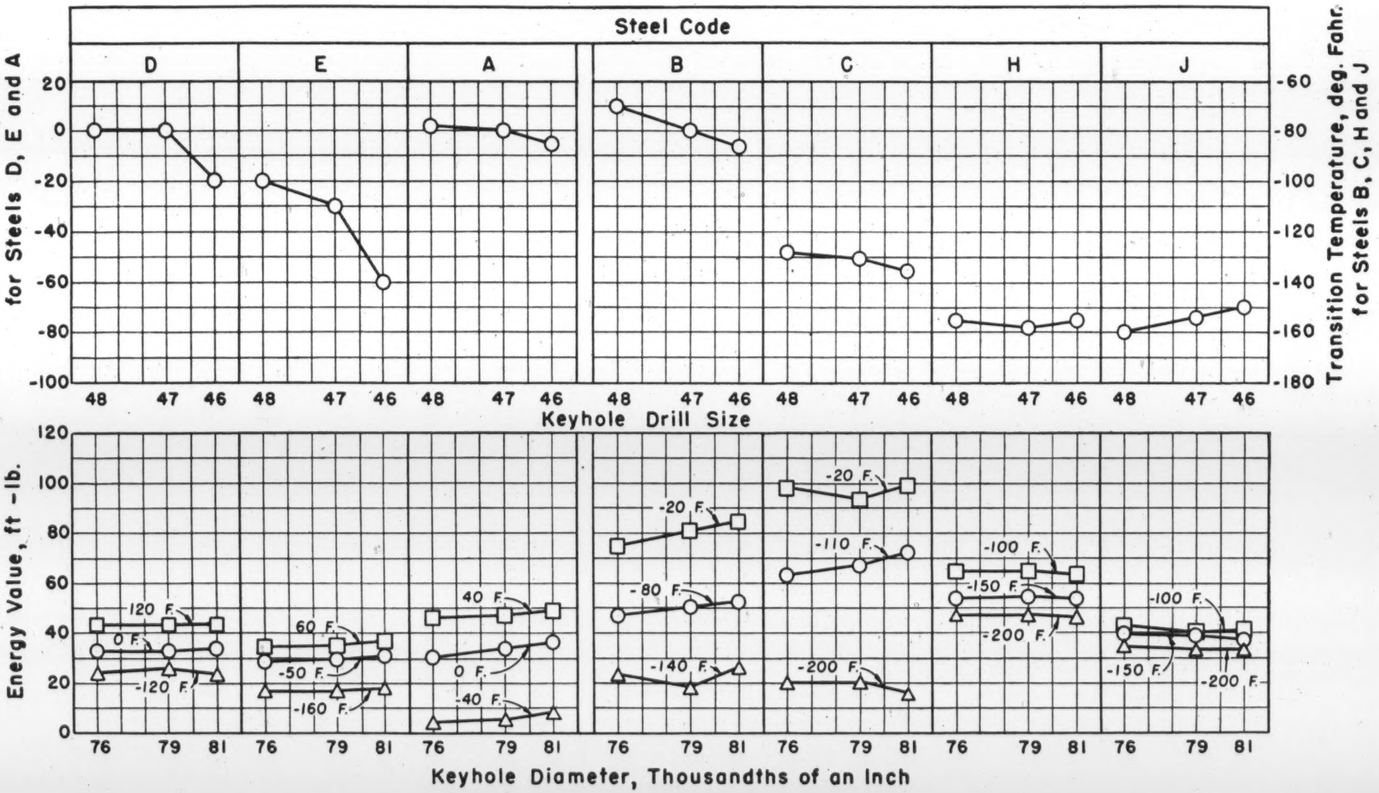


FIG. 13.—Effect of Keyhole Diameter on Transition Temperature and Energy Absorption Values.

temperatures of these steels. The authors are unable to offer any satisfactory explanation for this behavior, especially since they expected the keyhole notch to be relatively less sensitive than the V-notch to comparable variations in notch acuity.

Effect of Variations in Keyhole Diameter on Energy-Absorption:

As in the case of the V-notch, energy values were selected from the average energy-temperature curves at temperatures corresponding to high, intermediate, and low levels. The values have been plotted against the hole diameter in the lower portion of Fig. 13. It is to be observed that the energy values are only slightly sensitive to changes in keyhole-notch diameter ranging from 0.076 to 0.081 in.

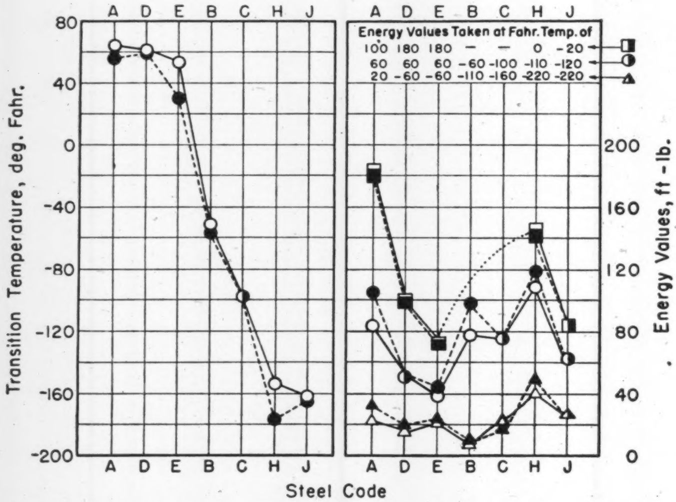


FIG. 14.—Effect of Polishing Root of Standard 0.010-in. Radius V-Notch on Transition Temperature and Energy Absorption Values.

Solid points are for polished notch; open points are for unpolished notch.

Effect of Polishing Root of Standard 0.010-in. V-Notch on Transition Temperature and Energy-Absorption:

The transition temperatures and energy absorption values as affected by polishing the root of the standard 0.010 in. radius V-notch are shown graphically in Fig. 14. In this graph, the steels have been arranged from left to right in order of decreasing transition temperature for the "as-machined" notch. In all cases, the transition temperature of the polished-notch specimens is the same as, or somewhat below, that of the unpolished notch. In general, it is to be noted that the effect of polishing on the transition temperatures and energy values is slight, as indicated by the close proximity of the polished and unpolished notch "curves."

Effect of Single versus Triple Cuts in Machining V-Notch:

In the work previously discussed, the V-notches were all cut to the required depth in three stages, namely, two roughing cuts and a final finishing cut. In the case of steels A and D, an additional series of standard 0.010-in. radius V-notch specimens was prepared in which the notch was cut to the required depth in one step, and tested in the "as-machined" condition and also after polishing the root of the notch. A comparison of the average energy-temperature curves obtained with single- and triple-cut V-notch specimens, polished and unpolished, is presented in Fig. 11 for steels A and D, respectively. The results obtained with single-cut notches, tested "as-machined" and also after polishing, were so closely alike that a single energy-temperature curve served to define the behavior in both conditions. This confirms previous indications that polishing the base of the V-notch appears to exert little influence on the test results. Figure 11 also indicates no pronounced differences in the behavior of these steels when the notch is cut in one step as compared to three cuts, judging by the close proximity of the various energy-temperature curves.

Suggested Tolerances for Specimen Dimensions:

1. *V-Notch Root Radius.*—The results of the investigation point to the need for applying tolerances to the root radius dimension of the standard V-notch which is currently specified in A.S.T.M. Method E 23 as 0.010 in. Since some steels are insensitive to a reduction in the root radius below approximately 0.011 in., the authors are of the opinion that the tolerance should be largely in the negative direction but that it should be limited to −0.002 in. rather than −0.005 in. as proposed by Bagsar (7) and that the tolerance in the positive direction should be relatively small. With a proposed specification for the notch root radius of 0.010 +0.0005, −0.002 in. the transition temperatures of the steels investigated would show a maximum deviation of approximately 10 F. from the value obtained with the 0.010-in. radius notch, on the basis of the average curves included in Fig. 12. In the case of insensitive steels, such as A, B, and C, the deviation would be practically negligible. Furthermore, it is evident from Fig. 12 that variations in the V-notch radius within the suggested tolerances would not appreciably affect the energy-absorption values.

2. *Hole Diameter in Keyhole-Notch.*—The results indicate that, when the diameter of the hole is 0.003 in. less than or 0.002 in. greater than the specified dimension of 0.079 in., the transition temperatures and, particularly, the energy-absorption values of the steels investigated do not, in general, deviate greatly from results obtained with the standard 0.079-in. diameter notch. In the case of one steel (Codes D and E), the increase in notch diameter appreciably affected the transition temperature without materially influencing the energy-absorption value. In view of the behavior of steel Codes D and E, the authors believe that the tolerance for the hole diameter should be largely in the negative direction and accordingly suggest that the diameter of the drilled hole be specified as 0.079 +0.001 −0.003 in.

3. *Section Thickness Beneath Notch.*—While the authors were concerned primarily with establishing tolerances for the standard V-notch root radius and keyhole diameter, they wish to comment on this specimen dimension in view of the fact that no tolerance is included in A.S.T.M. E 23 for the latter. It is believed that a tolerance should be applied to this dimension since one of the dimensions defining the fracture area, namely, the 0.394-in. width, carries a tolerance of ±0.001 in. For the section thickness beneath the notch, the authors wish to suggest that this dimension be specified as 0.315 ±0.001 in. for the V-notch specimen and 0.197 ±0.002 in. for the

keyhole-notch specimen. A somewhat wider tolerance has been indicated for the keyhole-notch since it is difficult to drill the hole without introducing some lack of parallelism between the axis of the hole and the bottom surface of the test piece.

SUMMARY

Data on the effects of small variations in notch root radius of standard Charpy V- and keyhole-notch specimens on the transition temperature and energy absorption level of a number of steels have been presented and discussed from the standpoint of establishing the need for dimensional tolerances, for which no standards exist at the present time. In addition, data have been included to illustrate the effects of polishing the root of the standard 0.010-in. radius V-notch and of machining this notch in single and multiple cuts.

The results of the investigation pointed to the need for specifying tolerances for the root radius dimension of the standard V-notch and the hole diameter dimension of the standard keyhole-notch. The authors have recommended that the root radius of the V-notch be specified as 0.010 +0.0005 −0.002 in. and the hole diameter in the keyhole-notch as 0.079 +0.001, −0.003 in. In addition, their experience in preparing approximately 4250 Charpy specimens for this investigation indicated that existing requirements for other dimensions of the Charpy specimen, as specified in A.S.T.M. Method E 23 - 47 T, are adequate and require no modification, with one exception, namely, the section thickness dimension beneath the notch for which no tolerances have been set. In this connection, the authors have recommended that the section thickness beneath the notch be specified as 0.315 ±0.001 in. for the V-notch and 0.197 ±0.002 in. for the keyhole notch.

In regard to polishing the root of the standard 0.010 in. radius V-notch, the data indicate, in general, no significant differences in transition temperature or energy absorption level between "as-machined" and polished notches, and the same was found to be true when the notch was machined to the required depth in a single cut as compared to multiple cuts.

Acknowledgment:

The authors wish to express their appreciation to the Bureau of Ships of the Navy Department for sponsoring this work and for permission to publish this paper. They are grateful to Capt. D. C. Redgrave, Jr., U.S.N., Director of the Material Laboratory New York Naval Shipyard, for his encouragement and review of the manuscript. The authors are greatly indebted to their associates, Messrs. V. Buglione, J. J. Gabriel, A. Meyerhoff, and Mrs. G. Fineberg for their aid in carrying out the experimental work and compiling the test data. They wish to thank Mr. G. A. Geyer, Master Mechanic of the Central Tool Shop of the New York Naval Shipyard, and his associates, Messrs. F. J. Kratchman and T. K. Pillsy, for their valuable contributions in the development and construction of jigs, fixtures, cutters and other equipment used in the machining of test specimens. Thanks are also due to Mr. F. C. Fyke and his co-workers of the Esso Engineering Dept. of the Standard Oil Co., for the information which served as a basis for the design and construction of the authors' equipment for conditioning Charpy specimens for tests at low temperatures.

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DISCUSSION

MR. JOSEPH H. SHAW.[1]—I should like to ask a question. What method was used in the killing of the steel? Was it silicon or aluminum killed?

MR. E. A. IMBEMBO (*author*).—All steels, except the medium steel, were fully killed with silicon. The medium steel was semi-killed.

MR. SHAW.—Was a residual silicon content specified?

MR. IMBEMBO.—It was not specified in the case of the medium and S.A.E. 1045 steels. The residual silicon content of these steels was approximately 0.04 and 0.22 per cent, respectively. The silicon content of the high-tensile and S.A.E. 4620 steels was specified to be in the range of 0.15 to 0.35 and 0.20 to 0.35 per cent, respectively.

MR. SAM TOUR.[2]—I think the authors should be congratulated on the quality of the work they have done. I should like to ask a couple of questions. One is with reference to using the highest possible velocity on the 220 ft-lb. impact machine. If you use the lower velocity as for the 110 ft-lb. capacity, would you find the same sensitivity or less sensitivity to these minor changes in notch acuity?

All of the steels tested had been heat treated to attain maximum impact properties at low temperatures. If the impact testing is on steels that are not so tough and with a higher hardness level, is it necessary to go to all these refinements?

That it is possible to machine a V-notch to 0.010 in. + 0.0005 in. − 0.002 in. does not constitute a necessity to hold to those close dimensions unless wide variations in results can be attributed directly to such dimensional variations.

Why specify such very close tolerance on depth of notch and size of keyhole when, as a matter of fact, the results have a wide spread irrespective of fine controls on dimensions?

MR. IMBEMBO.—In regard to Mr. Tour's first question, we were compelled to use the maximum striking velocity of 18.1 ft. per sec. corresponding to the 220 ft-lb. capacity since in four of the seven conditions of heat treatment, the V-notch specimens required breaking energies considerably above the 110 ft-lb. level. In the case of the keyhole-notch specimens, the energy values for all steels were below 110 ft-lb. and these specimens, therefore, could have been fractured at a lower striking velocity. However, for comparative purposes, it was considered desirable to use the same striking velocity throughout, and, accordingly, all tests were made under a velocity of 18.1 ft. per sec.

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[1] Engineering Dept., E. I. du Pont Nemours & Co. Inc., Wilmington, Del.

[2] Chairman of the Board, Sam Tour and Co., Inc., New York, N. Y.

In reply to Mr. Tour's second question, we did not heat treat the steels with a view of attaining maximum impact properties at low temperatures. The objectives of the heat treatment were to homogenize the steels for better reproducibility of results and to develop tensile strength levels ranging from 60,000 to 155,000 psi. which encompass the usual range of tensile strength encountered in constructional engineering steels. As it developed, some of the steels, when subjected to V-notch tests, exhibited very high energy-absorption levels at test temperatures above the transition region, requiring practically the full 220 ft-lb. capacity of the testing machine to fracture. Other steels showed V-notch energy-absorption values in the range of 75 to 150 ft-lb. when tested above the transition temperature region. However, our analysis of the data from the standpoint of establishing tolerances for the root radius of the V- and keyhole-notches was based on a consideration of the energy-absorption characteristics of the steels at temperatures above, within, and below the transition temperature region. In the case of the latter, we are in effect dealing with material of low toughness, the behavior of which might be considered comparable to those of "steels that are not so tough and with a higher hardness level".

With respect to steels of the type covered by our investigation, we believe that the data justify the establishment of the tolerances suggested by the authors for the root radius of the standard V- and keyhole-notches. It is believed that the exclusion of tolerances would result in rather wide variations in the energy values and transition temperatures.

MR. TOUR.—The authors have stated that they were working with impact values of approximately 200 ft-lb. That is not the normal impact value of structural steels. There are many structural steels in use with impact values on the order of 30 to 40 ft-lb. When dealing with the 30 or 40 ft-lb. impact value on Charpy tests, these fine variations may not have so large an effect. In fact, they do not. But it is proposed that in all impact testing in any range—not in the 100 ft-lb. or higher range, but in all ranges—specification of fine dimensions and controls be made.

It was stated that there are no specifications at present, yet there is a specification on one radius. There is no tolerance on that and the authors ask for +0.0005 and −0.002 in. tolerance on that radius. In addition they propose a very close tolerance on the depth of the notch.

They have not shown that the depth of the notch is of importance at all. When impact values drop to 30 ft-lb., the depth of notch has less effect.

MR. IMBEMBO.—The authors did not investigate the effects of variation in depth of metal beneath the notch. It was felt that since the A.S.T.M. has imposed a tolerance for the fracture width, certainly, by the same token, a tolerance should be applied to the other dimension, both of which define the fracture area. Otherwise, there would be no particular point in applying a tolerance to the width of the specimen.

THE FATIGUE STRENGTH OF STEEL THROUGH THE RANGE FROM $\frac{1}{2}$ TO 30,000 CYCLES OF STRESS*

By M. H. Weisman[1] and M. H. Kaplan[1]

Synopsis

Relatively few fatigue tests reported in the literature include data for lifetimes of less than 50,000 cycles. Data from these reports were compiled and have been replotted in terms of life *versus* stress in per cent of tensile strength. Uni-directional axial load and sheet bending fatigue tests were conducted to obtain short-life fatigue data for S.A.E. 4340 steel heat-treated to 215,000 psi. tensile strength. Fatigue strength curves based on the information compiled are presented for both smooth and notched specimens and for uni-directional as well as for reversed loading. The axial load *S-N* curve for the stress range zero to maximum stress is complete from $\frac{1}{2}$ cycle to the endurance limit. The *S-N* curves for other types of testing are complete from about 1500 cycles to the endurance limit.

The use of fatigue data, until recently limited to the analysis of service failures, is now finding recognition in the fields of aircraft, automotive and machine design. In these fields a more accurate knowledge of operating conditions combined with improved methods of stress analysis make it possible to design for a desired life expectancy.

Most often, in automotive and machine design the life expectancy will range from several hundred thousand cycles to many million cycles of critical stress. For the design of some types of military aircraft, however, the life expectancy of a number of components is less than 50,000 cycles of critical stress. Mechanisms such as those which extend landing gears, operate bomb bay doors or control wing flaps receive only limited usage during the entire life of an airplane. In these cases, to require an infinite life for maximum loading could mean an unnecessary size and weight penalty.

In the past, aircraft designs normally have afforded adequate fatigue strength. Reported fatigue failures have been few in proportion to all known aircraft failures. Current design trends, however, increase the possibility of fatigue failure by producing greater dynamic loading. Thus, the consideration of the effect of repeated stress is becoming an increasingly vital part of design **(19)**.[2] Though there is extensive information on the subject of fatigue available in the literature, it must be properly arranged and correlated before being useful for design purposes.

Relatively few fatigue tests reported in the literature include data for lifetimes of less than 50,000 cycles of stress. Hartman and Stickley **(1)** investigated the fatigue properties of the aluminum alloy 17S-T from $\frac{1}{2}$ cycle to 75,000 cycles of stress. A few reported fatigue

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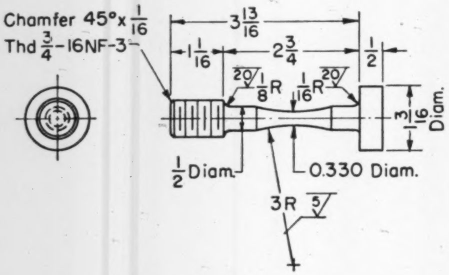
* This paper was presented at the Session on Fatigue of Metals held at the First Pacific Area National Meeting of the Society, San Francisco, Calif., October 10–14, 1949.

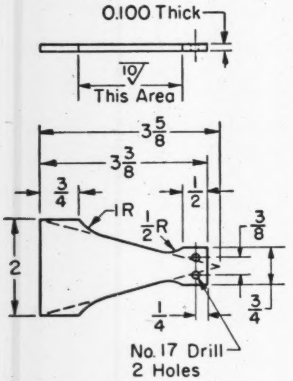
[1] North American Aviation, Inc., Los Angeles, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 664.

tests of steel include data for lifetimes as short as 10,000 stress cycles. Otherwise, most fatigue strength curves for steels begin in the region of 50,000 stress cycles and extend to the endurance limit beyond 10,000,000 stress cycles. It is believed that useful engineering information, particularly for application to aircraft design, would be provided by determining the characteristics of the short-life high stress portion of the *S-N* curve.



(*a*) Axial Load Specimen.



(*b*) Flexure Specimen.

FIG. 1.—Dimensions of Fatigue Test Specimens.

SCOPE

This paper presents a compilation of that portion of the published fatigue tests on steel which include data for lifetimes of less than 50,000 cycles.

In addition, test results which provide data for the range of life ½ to 30,000 cycles are presented for steels heat treated to 200,000 psi. tensile strength. Axial loading and flexural fatigue tests of unnotched specimens were conducted at the stress range $R = 0$; that is, from zero stress to maximum tensile stress.

DESCRIPTION OF SPECIMENS

The axial loading fatigue test specimens, Fig. 1(*a*), were machined from a 1¼-in. diameter bar of annealed S.A.E. 4340 steel. The steel was electric furnace aircraft quality produced to Army-Navy Aeronautical Specification AN-QQ-S-756a and Amendment No. 1, dated March 15, 1945. The flexural fatigue test specimens, Fig. 1(*b*), were machined from a $\frac{5}{16}$-in. thick plate of S.A.E. 4337 steel, produced to S.A.E. Aeronautical Material Specification AMS 6359, dated December 1, 1942. The chemical composition of the two steels is given in Table I and the physical properties in Table II.

The test specimens were rough machined within 0.030 in. of the finished dimensions and then heat-treated as follows: heated in a controlled atmosphere furnace at 1570 F. for 1 hr., oil quenched, then tempered in an air furnace at 800 F. for 2 hr. to the hardness given in Table II. During heat treatment, all specimens were suspended vertically in a fixture in order to minimize distortion. After heat treatment, the specimens were finish ground and hand polished. Final hand polishing of the reduced sections of both types of specimens was done with fine emery paper in such a manner as to eliminate any circumferential marks from the axial specimens and to break the sharp edges of the flexural fatigue specimens. All specimens were magnetically inspected prior to testing. No defects were found.

Axial loading test specimens were also machined from a 1⅜-in. diameter S.A.E. 4140 steel bar and from a 5 by 5-in. S.A.E. 4140 steel forged billet.

DESCRIPTION OF EQUIPMENT

Axial-Loading Tension Fatigue Tests:

The fatigue testing machine used for the axial-loading series of tests had been designed and constructed for the fatigue testing of high-strength bolts. This tester, shown in Fig. 2, was of the hydraulic type, with a ram area of 38.8 sq. in. and a rated capacity of 40,000 lb. The machine was driven by a 5-hp. electric motor coupled to a Vickers vane-type high-pressure hydraulic pump capable of delivering fluid at variable pressures up to 2000 psi. The on-and-off loading mechanism consisted of a Vickers Selector Valve operated by a ⅓-hp. Boston Ratiomotor geared down to a speed of 50 rpm. A mechanically-actuated counter recorded the number of load cycles. Automatic switches stopped the machine when a specimen failed. The operation of the selector valve was adjusted so that full loading and unloading were accomplished in each cycle without overtravel or surge while loading or undue impact during unloading.

TABLE I.—CHEMICAL COMPOSITION OF STEELS TESTED.

S.A.E. Steel	Form	Chemical Composition, per cent								
		Carbon	Manganese	Silicon	Sulfur	Phosphorus	Chromium	Nickel	Molybdenum	Copper
4340	1¼-in. diameter bar	0.41	0.74	....	....	....	0.82	2.00	0.30	....
4337	5/16-in. thick sheet	0.37	0.72	....	....	....	0.79	1.89	0.24	....
4140[a]	Forged billet	0.38	0.92	0.32	0.024	0.018	1.02	0.1	0.22	0.1
4140[a]	1⅜-in. diameter bar	0.40	0.79	....	0.019	0.025	0.97	....	0.22	....

[a] From paper by Van Camp (22).

TABLE II.—PHYSICAL PROPERTIES OF STEELS TESTED.

S.A.E. Steel	Tensile Strength, psi.	Yield Strength, 0.2 per cent off-set, psi.	Elongation in 2 in., per cent	Rockwell Hardness, C Scale
4340 bar........	215 700	210 400	....	43.5
4337[a] sheet.....	211 000	200 000	8.0	42.5
4140[b] billet......	193 500	183 500	12.0	42.0
4140[b] bar........	195 000[c]	....	....	42.5

[a] Axis of specimen perpendicular to direction of rolling.

[b] From paper by Van Camp (22).

[c] Tensile strength value converted from Rockwell hardness.

The machine was calibrated with special strain-gaged fixtures. It was found that load values could be computed with sufficient accuracy by multiplying the observed fluid pressure by the ram area and then correcting for the dead weight of the platens and fixtures. No hydraulic surges or inertial effects due to the weight of the platens were observed during the calibration tests.

The master test gage shown in Fig. 2 was used for calibrating and checking purposes but was not left on the machine during the course of the test.

Flexural Fatigue Tests:

The flexural fatigue tests were conducted on a specially designed machine with which repeated-bending loads were imposed on the specimen by dead weights through a cam-actuated cable system. The cable system, shown in Fig. 3 was composed of a harness and a main cable which joined the harness to the weight pan. The harness consisted of a central chain and two external flexible cables, the terminals of which were securely attached to two steel yokes. On the forward stroke of the cam, the external flexible cables slackened and the full load of the weights was applied to the specimen through the central chain; on the backward stroke, the external cables lifted the weights until the specimen was completely unloaded as evidenced by the slackening of the central chain. In this manner, the full load of the weights was

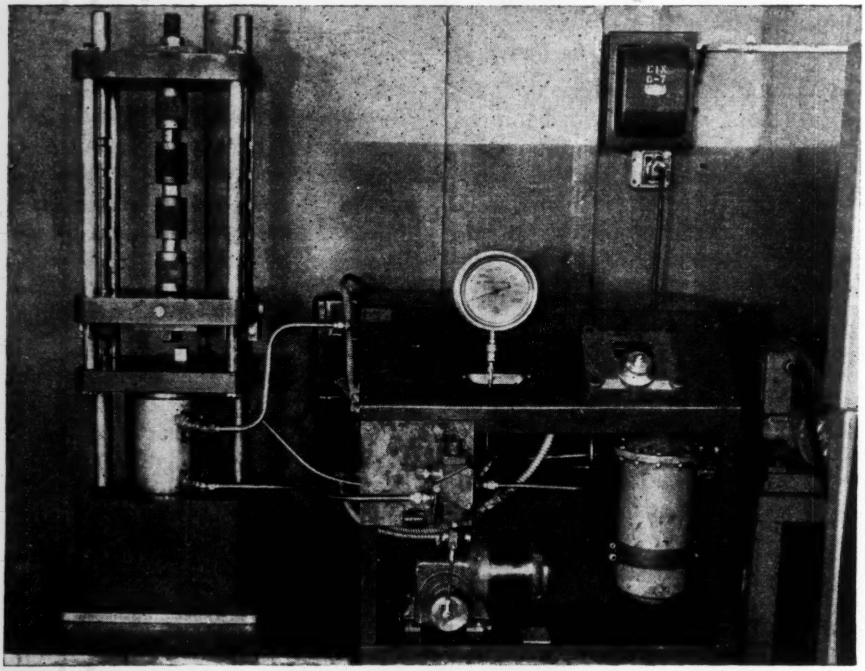


FIG. 2.—Axial Load Fatigue Machine.

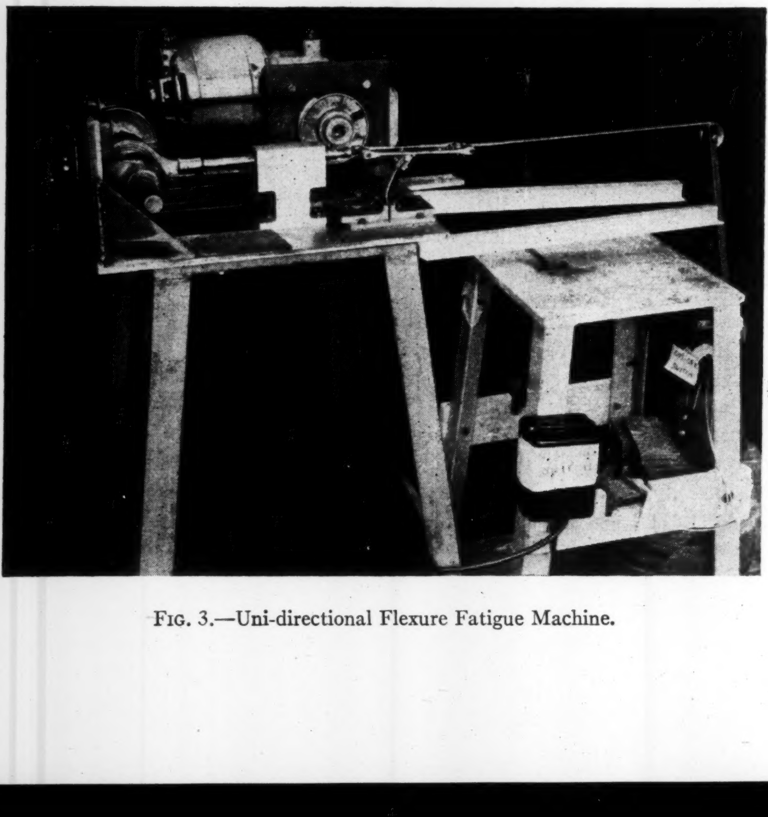


FIG. 3.—Uni-directional Flexure Fatigue Machine.

alternately applied and then removed from the specimens during each rotation of the cam even though permanent deformation of the specimen had taken place. The cam was driven by a $\frac{1}{3}$-hp. Boston Ratiomotor geared down to a speed of 35 cpm. A mechanically-actuated counter recorded the number of load cycles and an automatic switch stopped the machine when the specimen failed.

Test Procedure

Tension Tests:

The static tension test of the S.A.E. 4340 steel was made on one of the specimens prepared for the axial load fatigue tests. Tension test specimens for the S.A.E. 4337 steel were machined from the base sections of two flexural fatigue test specimens. A 20,000-lb. capacity Riehle testing machine was used for these tests. Yield strength was determined from the stress-strain diagram by the 0.2 per cent offset method.

Axial Load Fatigue Tests:

The axial load fatigue tests were performed on the bolt testing machine (Fig. 2) described previously. Special fixtures were used to insure specimen alignment during testing. The maximum stress level for the first test was 211,200 psi., or 98 per cent of the tensile strength of the material. The stress range for all tests was from zero to maximum tensile stress. Thereafter, the maximum stress was progressively reduced for each successive test so as to obtain adequate coverage of the life range under investigation. Test Nos. 3 and 4 were both run at approximately the same stress level in order to check the reproducibility of the results.

Flexural Fatigue Tests:

Six fatigue specimens were tested on the flexure fatigue machine (Fig. 3) described earlier. The specimens were subjected to repeated uni-directional bending loads, the maximum outside fiber stresses applied ranged from 186,000 to 208,500 psi. No reversal of stress was applied; that is, the specimen was loaded to the maximum stress and then allowed to return to zero stress before being loaded again in the same direction. Complete removal of the load during each cycle permitted the specimen to return to zero stress, despite varying amounts of permanent deformation.

The flexural fatigue specimens were mounted securely to the fatigue machine by means of two angle plates. A special loading fixture was bolted to the top of each specimen. The load was applied to the specimens by a cable system, one end of which was attached to a pin in the loading fixture and the other end attached to the weights (Fig. 3).

The calculated maximum outer fiber stress, uncorrected for plastic bending, for the first flexural fatigue test specimen was 264,500 psi. When corrected for bending in the plastic range by the method of Cozzone **(24)** (using a shape factor of 1.5), the maximum stress (S_B) was 186,000 psi., or 88.1 per cent of the ultimate tensile strength. Test No. 2 was run at the same stress level to check the reproducibility of the results. The test stress level was increased to 191,500 psi. (275,000 psi. calculated) for test No. 3 and to 195,500 psi. (281,500 psi. calculated) for test No. 4. Test No. 5 was run at 198,500 psi. (286,000 psi. calculated) to check the results of test No. 4. Test No. 6 was run at a stress level of 208,500 psi. (298,500 psi. calculated).

The test stress values noted as "calculated" were determined by the equation

$$S = \frac{Mc}{I} \qquad \ldots\ldots\ldots\ldots (1)$$

corrected for errors (other than plastic bending) due to the deflection of the specimen.

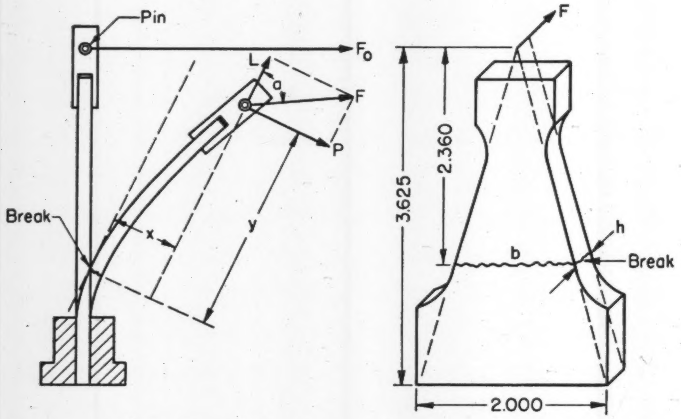


FIG. 4.—Layout for Calculation of Corrected Stress for Flexure Fatigue Tests.

TABLE III.—RESULTS OF FATIGUE TESTS OF STEEL SPECIMENS LOADED AXIALLY FROM ZERO TO MAXIMUM TENSION STRESS (R = 0).

Specimen	S.A.E. Steel	Maximum diameter, in.	Load, lb. (±75 lb.)	Stress, psi.	Percentage of Tensile Strength	Cycles to Failure
No. 1	4340	0.330	18 450	215 700	100.0	½[a]
No. 2	4340	0.332	18 250	211 200	97.9	10
No. 3	4340	0.328	17 400	206 000	95.5	248
No. 4	4340	0.324	16 900	205 000	95.1	226
No. 5	4340	0.320	17 500	202 100	93.8	1 968
No. 6	4340	0.332	17 100	197 500	91.7	6 517
No. 7	4340	0.329	16 500	194 100	90.0	9 770
No. 8	4340	0.327	16 000	190 500	88.4	11 476
No. 9	4340	0.327	15 500	184 600	85.6	17 816
No. 10	4340	0.333	15 000	172 200	79.6	32 661
No. FO[d]	4140[b]	0.331	13 290	154 200	80.0	30 845
No. FI[d]	4140[b]	0.331	13 290	154 200	80.0	19 539
No. BS6[d]	4140[c]	0.331	13 290	154 200	80.0	37 468
No. BS6L[d]	4140[c]	0.331	13 290	154 200	80.0	30 379

[a] Tensile test specimen.
[b] Longitudinal specimens from forged billet.
[c] Specimens from 1⅜-in. diameter bar.
[d] From paper by Van Camp (**22**).

If the specimen deflection was small, weights of 257.5 lb., 275.9 lb., and 303.4 lb. would produce maximum calculated outer fiber stresses of 280,000 psi., 300,000 psi., and 330,000 psi., respectively. The specimen deflection was of such magnitude, however, as to render inaccurate calculations based solely on Eq. 1. The method of calculating the deflection correction is given below.

The calculated outer fiber stress for specimen Nos. 1, 4, and 6 was determined in the following manner. A piece of white paper with a backing support was placed alongside the specimen and a tracing was made of the edge outline in the no-load position, Fig. 4. Weights of 257.5, 275.9, and 303.4 lb. were placed on the load pan in this order, and a tracing of the deflection curve in the specimen outline was made for each weight position. The accuracy of the tracings was checked by direct measurement of the horizontal and vertical movement of the loading pin for each load position, Fig. 4.

Using the tracings, a line was constructed tangent to the outer (tension) side of the specimen at the break point (Fig. 4). One component, L, of the load, F, was drawn parallel to the tangent line, and the other component, P, was drawn perpendicular to the tangent line. The direction of application of the force, F, was drawn for each load by constructing a straight line from the specimen loading pin to the cable-support pulley of the fatigue machine (Fig. 3). The distances, x, and y, and the angle, a, were then determined from the tracing by measurement. The corrected bending moment at the location of failure, M_b, due to the force, F, is the summation of the moments of the components, L and P:

$$M_b = M_P - M_L$$
$$M_b = y\,F \sin a - (x\text{-}c)\,F \cos a$$

where:

y = moment arm of component P and
x-c = moment arm of component L to neutral axis of specimen.

The corrected bending stress ($S_{\max}$) is

$$S_{\max} = \frac{M_b c}{I}$$

$$S_{\max} = [yF \sin a - (x - c)F \cos a]\left[\frac{c}{I}\right] \quad (2)$$

The location of failure of all specimens was approximately the same distance, $1\frac{17}{64}$ in., from the base of the specimen. At this location the width of the specimen was 1.30 in. and the cross-sectional area, A, was 0.130 sq. in. The resulting section modulus was:

$$\frac{I}{c} = \frac{bh^2}{6} = 0.00217 \quad (3)$$

Therefore,

$$S_{\max} = \frac{F[y \sin a - (x - c) \cos a]}{0.00217} \quad (4)$$

Finally, the axial tension stress, S_T due to the component of force, L, parallel to the specimen at the break: $S_T = \frac{L}{A}$ was added to $S_{\max}$ to obtain the maximum corrected outer fiber stress, S_b, at the point of failure:

$$S_b = S_{\max} + S_T$$

Therefore,

$$S_b = \frac{F[y \sin a - (x - c) \cos a]}{0.00217} + \frac{F \cos a}{A} \quad (5)$$

RESULTS

The results of the axial-loading fatigue tests at loads of 80 to 100 per cent of the static tensile strength are presented in Table III and are plotted in Figs. 6 and 9. The results of the sheet bending fatigue tests at loads of 88.1 to 98.9 per cent of the static tensile strength are presented in Table IV and are plotted in Figs. 6 and 7. The specimens tested in the axial-loading fatigue tests and in the flexural fatigue tests are shown in Figs. 11 and 12, respectively.

A review of the literature disclosed few reports of fatigue tests of steel which presented data for fatigue lifetimes of less than 50,000 stress cycles. The data from these few reports were compiled and are presented in Figs. 5 through 10 in terms of life *versus* stress in per cent of static tensile strength, rather than life *versus* actual applied stress. By this means, it is possible to compare separate tests wherein the test method and heat treatment differ. Curves based on the information compiled are presented for both smooth and notched specimens, and for uni-directional as well as completely reversed loading. The experimental data obtained in this investigation are plotted with corresponding test data from the literature. In the case of the axial loading tests, the resultant curve is complete from the static tensile strength to the endurance limit for the stress ratio of zero ($R = 0$). The curves for the other types of testing are complete from about 1500 cycles to the endurance limit.

DISCUSSION

The primary object of this investigation was to determine the shape of the S-N curves for steel for the life range

$\frac{1}{2}$ to 30,000 cycles of stress. It was felt, however, that presentation of the *S-N* curves complete to the endurance limit would aid in interpretation of the results.

The presentation of fatigue data in this paper in terms of life *versus* stress in per cent of tensile strength permits comparison of test results for steels of different tensile strength values and different heat treatments. Thus, annealed, normalized, and hot-finished steels can be compared with quenched-and-tempered steels.

The authors believe that any conclu-

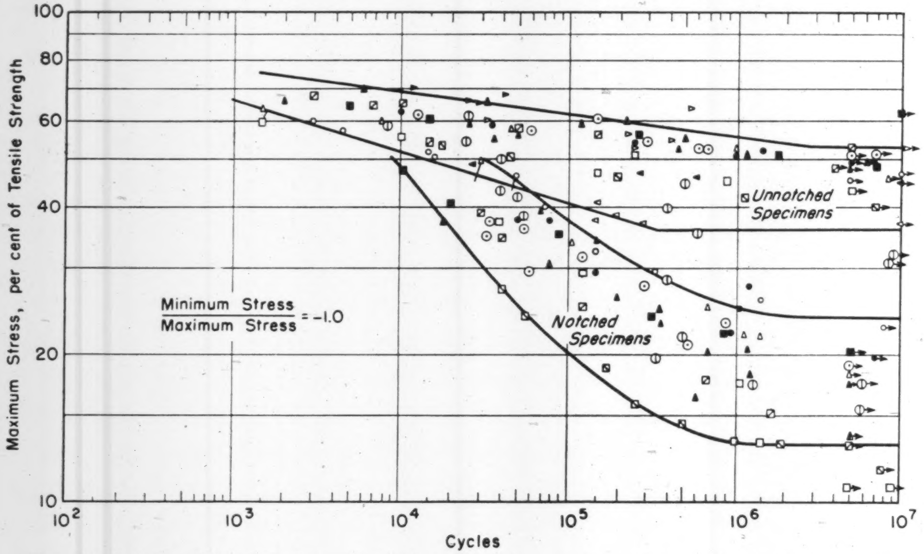


FIG. 5.—Relationship of Fatigue Strength to Life from a Compilation of Axial Loading Test Results (Reversed Stress, $R = -1.0$).

(Refer to Table V for tensile properties of steels tested.)

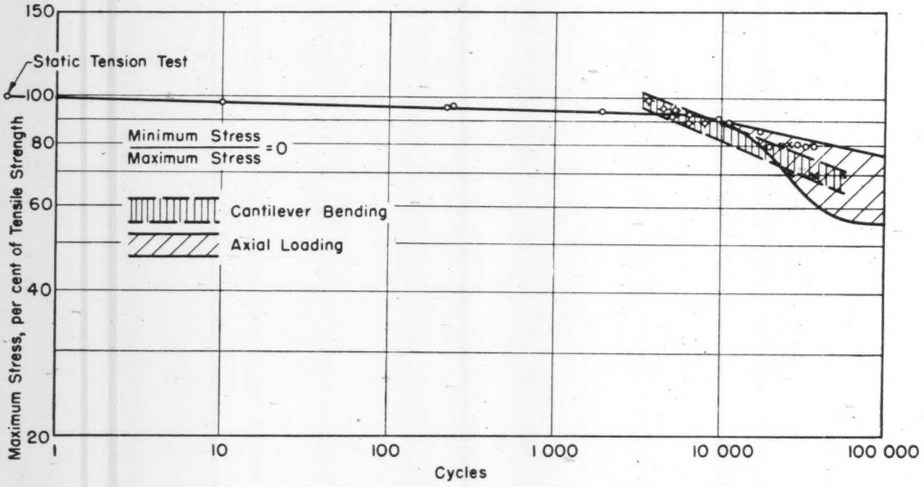


FIG. 6.—Comparison of Uni-directional Axial Loading and Flexure Fatigue Tests.

sions based on the type of data compiled in this paper can be only of a general nature. It must be remembered that laboratory fatigue tests are designed to obtain accurate and reproducible measurements under carefully restricted conditions. Consequently, an engineer attempting to evaluate fatigue properties for a particular problem may find much data but no single value which fits exactly the specific use he has in mind. Laboratory fatigue data must be correlated with service data before the results can be accepted for design use.

TABLE IV.—RESULTS OF SHEET-BENDING FATIGUE TESTS OF S.A.E. 4337 STEEL SPECIMENS UNDER UNI-DIRECTIONAL LOADING ($R = 0$).

Specimen	Load, F, lb.	Uncorrected Calculated Bending Stress, $S = \frac{Mc}{I}$, psi.	Corrected Calculated Bending Stress, S_b, psi.	Corrected Maximum Outer Fiber Stress, S_B, psi.	Ratio of S_B to Tensile Strength, per cent	Cycles to Initial Crack	Cycles to Final Failure
No. 1	257.47	280 000	264 500	186 000	88.1	....	8125
No. 2	257.47	280 000	264 500	186 000	88.1	6000	6450
No. 3	266.68	291 000	275 000	191 500	90.8	4200	5000
No. 4	275.86	300 000	281 500	195 500	92.7	4400	5330
No. 5	280.76	306 000	286 000	198 500	94.0	4150	4640
No. 6	303.45	330 000	298 500	208 500	98.9	500	3800[a]

[a] Test stopped before specimen failed completely.

TABLE V.—TENSILE PROPERTIES FOR DATA PLOTTED IN FIG. 5. COMPILATION OF AXIAL LOADING FATIGUE TESTS ($R = -1.0$).

$$\frac{\text{Minimum Stress}}{\text{Maximum Stress}} = -1.0$$

Symbol	S.A.E. Steel	Form	Tensile Strength, psi.	Yield Strength, 0.2 per cent offset, psi.	Elongation in 2 in., per cent	Hardness	Type of Notch	Reference
	4120	20-mm. Diameter Bar	87 000	72 000	29.6	181 BHN	60-deg. V-Notch 0.020-in. deep, 0.004-in. root radius	
	4120		114 000	95 000	21.8	234 BHN		
	4120		136 000	121 000	18.7	278 BHN		
	4130	30-mm. Diameter Cast Bar	131 000	112 500	13.8	273 BHN		(7)
	6130	30-mm. Diameter Bar	92 000	59 000	29.5	195 BHN		
	6130		108 500	98 000	22.8	232 BHN		
	6130		140 000	126 000	18.5	300 BHN		
	6150	30-mm. Diameter Bar	131 000	114 500	18.7	269 BHN		
	6150		131 500	119 000	20.3	279 BHN		
	6150		198 000	184 000	10.9	399 BHN		
	5135	30-mm. Diameter Bar	140 000	123 000	17.3	296 BHN		
	HT-50	⅞-in. Diameter Bar	66 500 (As rolled)	48 600	32.0	135 BHN	None	(3)
	4340	¾-in. Diameter Bar	150 400	133 100	18.2	322 BHN		
	1045	Unknown	102 000	....	....	207 BHN	None	(14)
	1072	Unknown	168 500	....	....	332 BHN		

There is often a failure to recognize that the fatigue strength as determined for a particular specimen is not an inherent property of the material but is a function of the testing method and the dimensions of the piece as well as its history, that is, heat treatment and method of fabrication. Most investigations have reported no "speed effect" on fatigue test results for cycling speeds up to 10,000 cycles per minute **(3,10)**.

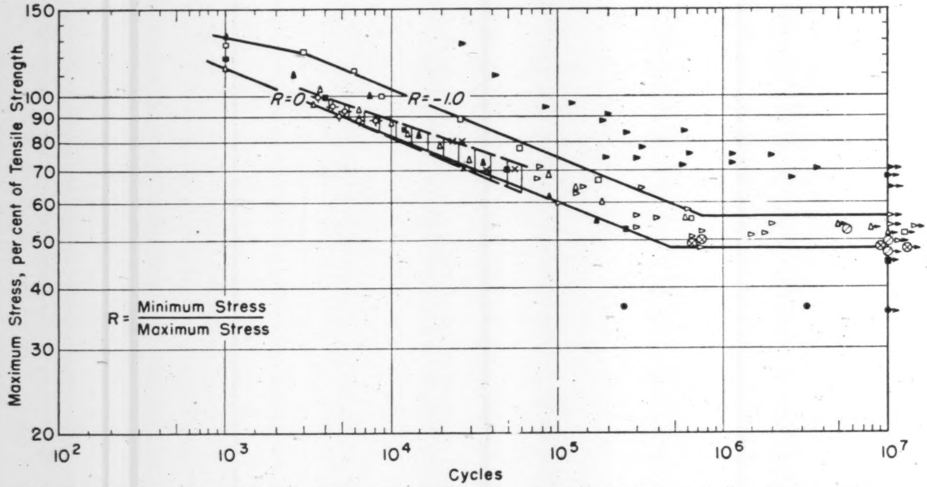


FIG. 7.—Relationship of Fatigue Strength to Life from a Compilation of Flexure Fatigue Tests.

(Refer to Table VI for Tensile Properties of Steels tested.)

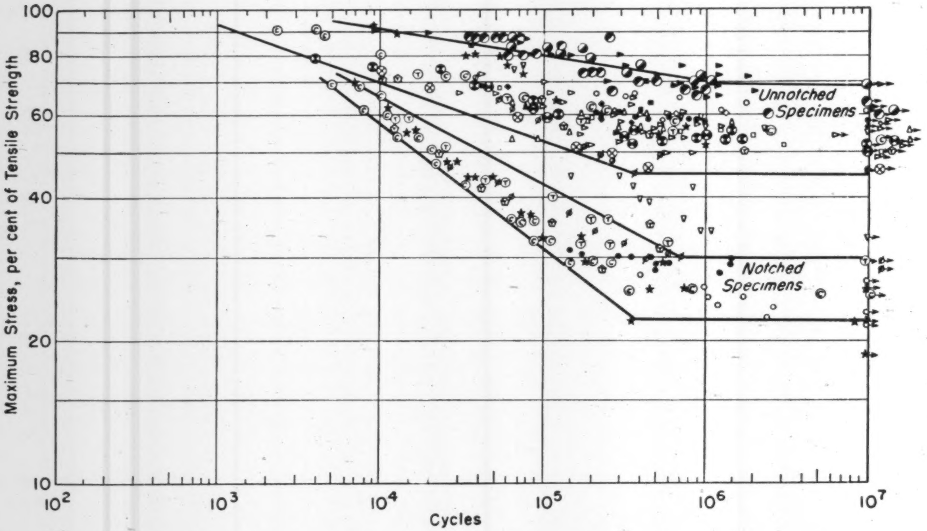


FIG. 8.—Relationship of Fatigue Strength to Life from a Compilation of Rotating Beam Test Results.

(Refer to Table VII for Tensile Properties of Steels Tested.)

This conclusion has been questioned by Wishart in a discussion of the paper by Oberg and Johnson **(10)** and by Koos, Lemmon, and Ransom **(21)**. Recent work for the National Advisory Committee for Aeronautics indicates that slow speeds of testing (below 100 cpm.), such as are most suitable for short-life tests, give lower fatigue strengths for normalized steels than do the more common speeds of several thousand cycles per minute. No speed effect was shown for 24S-T3 and 75S-T6 aluminum alloys, tests at low and high speeds giving approximately the same results. For the above reasons the following comments on the short-life portion of the *S-N* curves are confined only to general trends.

TABLE VI.—TENSILE PROPERTIES OF DATA PLOTTED IN FIG. 7. COMPILATION OF CANTILEVER BENDING (FLEXURE) FATIGUE TESTS.

Symbol	S.A.E. Steel	Form	Tensile Strength, psi.	Yield Strength, 0.2 per cent off-set, psi.	Elongation in 2 in., per cent	Hardness	Type of Notch	Reference
			UNI-DIRECTIONAL FLEXURE (ZERO TO MAXIMUM STRESS, $R = 0$)					
	4337	⁵⁄₁₆-in. thick plate	210 000	....	....	42.5 R_c	None	Table III
	9260	¼ x 1⅛-in. plate	Approximately 200 000	....	....	40–45 R_c	None, as rolled surface	**(16)**
			REVERSED FLEXURE ($R = -1.0$)					
	4130	⅜-in. thick plate	129 000	118 000	17.0	26 R_c	None	**(4)**
	4130		150 000	143 000	14.5	34 R_c		
	4130		206 000	194 000	10.0	44 R_c		
	2330	¾-in. diameter bar	108 600	88 700	12.5	246 VPH	None	**(13)**
	2330		145 300	139 300	17.5	322 VPH		
	8630		89 800	80 500	16.0	217 VPH		
	8630		142 000	121 500	15.5	302 VPH		
	HT-50	⅞-in. diameter bar	66 500 (As rolled)	48 600	32.0	135 BHN	None	(3)
	4340	¾-in. diameter bar	150 400	133 100	18.2	322 BHN		

Previously published data for axial loading fatigue tests for the stress range $R = 0$ **(3)** defined the life span from 50,000 cycles to the endurance limit. The data determined in this investigation extended the curve backward to $\frac{1}{2}$ cycle life, that is, the static tension test. This newly defined portion of the curve connects rather well with the previously known portion of the curve. However, it should be noted that the fatigue life at stresses above 80 per cent of the tensile strength is greater than would be indicated by extrapolation of the *S-N* curve determined at lower stresses. Hartmann and Stickley **(1)** make the same observation concerning the *S-N* curve for 17S-T.

The uni-directional flexure fatigue data (stress range $R = 0$) are not sufficient to indicate the shape of the *S-N* curve. The band shown in Figs. 6 and 7 connects the data from reference **(16)** with data determined in this investigation. These data agree very well with the data for the uni-directional axial loading tests. The *S-N* curve for reversed flexure (stress range $R = -1.0$) is well defined for the life span of 2000 cycles to the endurance limit. So also are the curves for reversed stress axial loading and rotating-beam tests. In general, steels quenched and tempered to tensile strengths less than 180,000 psi. have a higher ratio of fatigue strength to tensile strength than do steels of higher hardness. This is most evident in the results of the reversed flexural fatigue tests (Fig. 7).

For all three types of fatigue tests, the reduction of fatigue strength due to notches, that is, the fatigue strength reduction factor, becomes smaller as the test stress increases and the fatigue life decreases. In fact, it is probable that the *S-N* curve for notched specimens does not merge with but crosses the curve for unnotched specimens, so that the notched specimens actually develop a greater apparent fatigue strength than

TABLE VII.—TENSILE PROPERTIES FOR DATA PLOTTED IN FIG. 8. COMPILATION OF ROTATING BEAM FATIGUE TESTS.

Symbol	S.A.E. Steel	Form	Tensile Strength, psi.	Yield Strength, 0.2 per cent offset psi.	Elongation in 2 in., per cent	Hardness	Type of Notch	Reference
	4140	Forging (Longitudinal)	136 650	88 150	13.4	323 BHN	Kommers' square type notch	(2)
	4140	Forging (Transverse)	141 770	106 800	11.7	323 BHN		
	4140	Casting (Longitudinal)	138 500	95 250	13.2	324 BHN		
	4140	Casting (Transverse)	137 700	94 300	11.7	324 BHN		
	4130	⅜-in. thick plate (Parallel to Direction of rolling)	129 000	118 000	17.0	26 R_c	None	(4)
	4130		150 000	143 000	14.5	34 R_c		
	4130		206 000	194 000	10.0	44 R_c		
	4340	Unknown	150 400	142 000	18.2	....		
	A.S.T.M. A7-46	½-in. thick plate	64 200	42 600	27.7	....	None	(6)
	4340	3-in. diameter bar	150 000	142 000	18.2	....		
	4130	Unknown	125 000	99 400	15.9	....	None	(8)
	4340	3-in. diameter bar	163 700	157 200	16.1	36 R_c	Semi-circular notch 0.020-in. radius 0.025-in. deep, 0.250-in. root diameter	(9)
	4130	Rolled Bar	76 200	45 000	32.0	145 BHN	60-deg. V-notch 0.025-in. deep by 0.010-in. root radius, 0.250-in. root diameter	(10)
	4130	Rolled Bar	139 500	120 000	18.0	290 BHN		
	4130	Rolled Bar	199 300	168 500	10.0	432 BHN		
	x4340	Unknown	162 000	Unknown	Unknown	34 R_c	None, smooth machined finish	(12)
	x4340	Unknown	162 000	Unknown	Unknown	34 R_c	None, super-polish finished	
	2330	¾-in. diameter bar	108 000	88 700	12.5	246 VPH	None	(13)
	1045	Unknown	102 000	Unknown	Unknown	207 BHN	None	(14)
	1072	Unknown	168 500	Unknown	Unknown	332 BHN	None	
	6130	....	137 600	....	....	....	0.065 to 0.250-in. radius semi-circular grooves	(15)
	6130	....	137 600	....	....	....	0.010 to 0.015-in. root radius, "V" notches	
	HT-50	⅞-in. diameter bar	66 500 (As rolled)	48 600	32.0	135 BHN	None	(3)
	4340	¾-in. diameter bar	150 400	133 100	18.2	322 BHN		

the unnotched specimens for some lifetime less than 1000 cycles of stress. It is well established that a notched steel bar has a considerably higher ultimate tensile strength in the notched area than has a regular tension specimen (18). The increase in tensile strength of the notched bar results from the increased resistance of the metal to deformation, produced by the triaxial stress condition prevailing at the base of the notch. This same condition limits the ability of the notched fatigue specimen to strain harden, decreases the fracture stress and so pro-

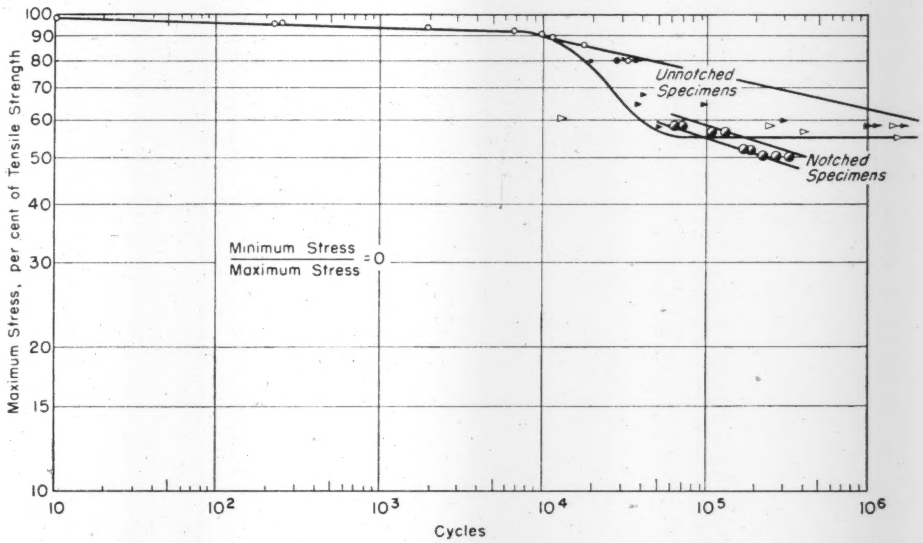


FIG. 9.—Relationship of Fatigue Strength to Life from a Compilation of Axial Loading Test Results (Stress Range from Zero to Maximum Tension, $R = 0$).

(Refer to Table VIII for Tensile Properties of Steels Tested.)

TABLE VIII.—TENSILE PROPERTIES FOR DATA PLOTTED IN FIG. 9. COMPILATION OF AXIAL LOADING FATIGUE TESTS (R = 0).

$$\frac{\text{Minimum Stress}}{\text{Maximum Stress}} = 0.$$

Symbol	S.A.E. Steel	Form	Tensile Strength, psi.	Yield Strength (0.2 Per-cent Off-set), psi.	Elongation in 2 in., per cent	Hardness	Type of Notch	Reference
	HT-50	⅞-in. Diameter Bar	66 500 (As rolled)	48 600	32.0	135 BHN	None	(3)
	4340	¾-in. Diameter Bar	150 400	133 100	18.2	322 BHN	None	
	A.S.T.M. A7-46	½-in. Thick Plate	64 200	42 600	27.7	....	1-in. diameter hole in center of 3½ by ½-in. section	(6)
	4340	1¼-in. Diameter Bar	215 700	210 400	....	43.5 R_c	None	Table II
	4140	5 x 5-in. forged Billet	193 000	183 000	12.0	42 R_c	None	
	4140	1⅜-in. Diameter Bar	195 000	....	....	42.5 R_c	None	

duces the rapid decrease in notched fatigue strength with increasing life.

The scatter bands for the different types of testing (Figs. 5, 7, 8 and 9 (Tables III to VIII)) have been replotted together for the stress ranges $R = 0$ (Fig. 6) and $R = -1.0$ (Fig. 10) for the life range $\frac{1}{2}$ to 100,000 cycles. The following comparisons are made concerning the effect of type of test on the results.

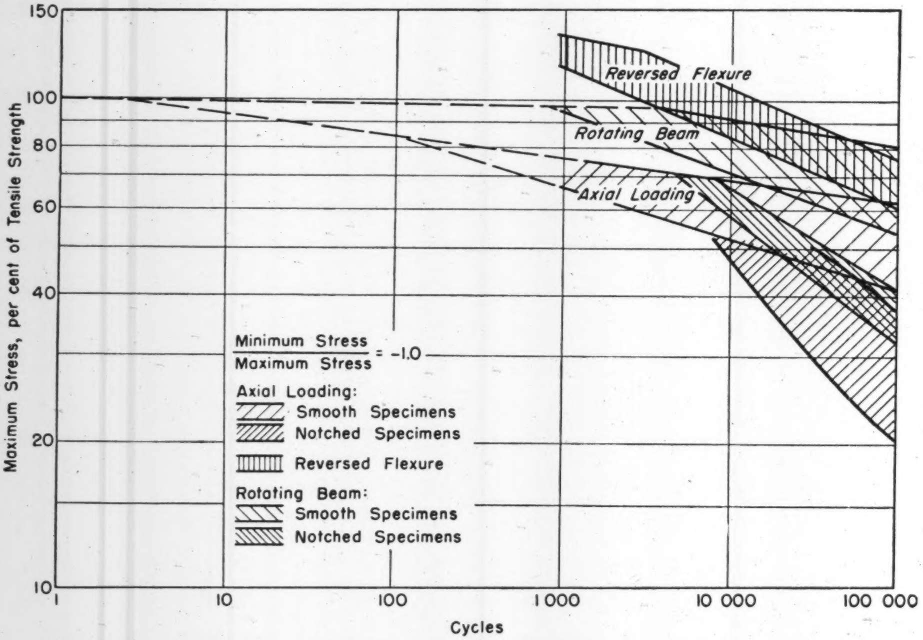


FIG. 10.—Comparison of Reversed Stress Axial Loading, Flexure, and Rotating-Beam Fatigue Tests.

The reversed flexure tests show higher fatigue strengths at lifetimes of less than 50,000 cycles than do the rotating beam tests. Some of this increase would probably be reduced if the results were corrected for the effect of plastic bending, since the test stresses in this part of the *S-N* curve are usually above the yield strength of the material. At longer lifetimes the scatter bands for the two types of tests merge. Some authors, however, are of the opinion that flexure tests produce higher results than rotating-beam tests over the entire life span **(4, 20, 21)**.

Reversed stress axial loading tests appear to give lower results over the entire life span than do either the rotating-beam or reversed-flexure tests.

The most common flexural fatigue tests are of the constant amplitude type, where the travel, or deflection, of the specimen is constant regardless of the specimen stiffness. Such tests are satisfactory for only a limited number of applications, since specimen stiffness usually decreases under conditions of repeated stressing. This decreasing stiffness in service usually results in an increased amplitude of movement and an acceleration of fatigue failure of the part. To reproduce this condition for constant amplitude machines involves continual readjustment of the amplitude of deflection as the stiffness of the specimen changes during testing.

The uni-directional flexure fatigue

test method presented in this paper provides a constant-stress type of test. Variations in the specimen deflection due to changes of elasticity of the material during testing do not introduce into the test the errors of the constant amplitude test outlined in the preceding paragraphs. The magnitude of specimen deflection which made a correction of the applied stress necessary in this investigation, can be considerably reduced by use of a thicker specimen. It is believed that this constant-stress testing method can be very useful for investigations of the high-stress low-cycle portion of *S-N* curves at other than room temperatures. Since the specimen is vertical there are no moving parts which would require sealing where liquid cooling media are used.

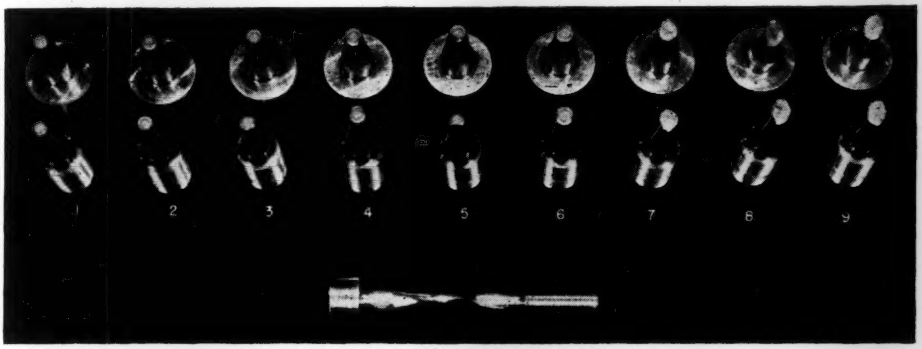


FIG. 11.—Axially Loaded Tension Fatigue Specimens Showing Failures.

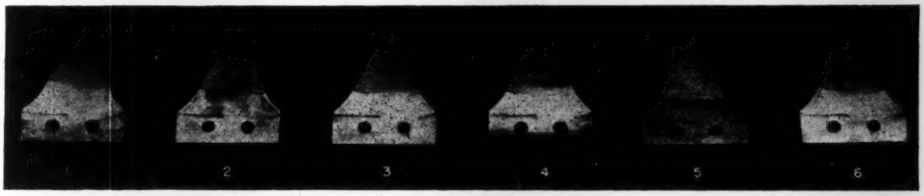


FIG. 12.—Flexure Fatigue Specimens Showing Failures.

SUMMARY

1. An *S-N* curve complete from $\frac{1}{2}$ cycle (static tension test) to the endurance limit has been drawn from data for uni-directional axial tension fatigue tests. The newly defined short-life portion of the curve shows that the fatigue life at stresses above 80 per cent of the tensile strength is greater than would be predicated by extrapolation backward of the long-life portion of the *S-N* curve.

2. The *S-N* curves for reversed bending, reversed axial loading, and rotating-beam fatigue tests are well defined over the range 2000 cycles to the endurance limit.

3. The bending fatigue test method presented in this paper provides a constant-stress flexure fatigue test which should be very useful for experimentation at other than room temperature.

4. Steels quenched and tempered to tensile strengths less than 180,000 psi. have a higher ratio of fatigue to tensile strength than do steels of higher hardness.

5. The reduction of fatigue strength

due to stress concentrations becomes less as the test stress increases and the fatigue life decreases. It is probable that the S-N curve for notched specimens crosses the S-N curve for smooth specimens and that the notched specimens will have greater fatigue strength than unnotched specimens for some lifetime in the range of less than 1000 cycles of stress.

RECOMMENDATION FOR FUTURE WORK

Much more work must be done on the following items before low-cycle fatigue data can be effectively used in design:

1. Effect of heat treatment and metallurgical structure.
2. Speed effect for cycling rates of less than 100 cycles per minute.
3. Effect of type of test and specimen shape.
4. Fatigue stress concentration factor for notches.

Acknowledgment:

The authors wish to express their thanks to N. B. Green of the Production Development Laboratory, North American Aviation, Inc., who conducted the axial load fatigue tests. The authors are indebted to L. P. Spalding and F. B. Bolte, Chief Research Engineer and Assistant Chief Research Engineer, respectively, and to F. R. Kostoch, North American Aviation, Inc., for their constructive interest in this investigation, and to the company for permission to publish this paper.

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DISCUSSION

MR. T. E. PIPER[1] (*presented in written form*).—In general, the investigation of the low-N part of the S-N curve is a commendable work long overdue.

The authors invite criticism of the conventional test procedures employed. Although the endurance limits of notched and un-notched specimens are recognized as almost academic values in terms of design criteria, I feel strongly that superimposed $-N$ of high frequency on $-N$ of low frequency introduces fatigue characteristics of a significantly deleterious degree. The "speed effect" should be treated as a complex characteristic of aircraft rather than a simple S-N property.

MR. A. E. ZEZULA.[2]—The authors are to be congratulated for the aggressive and progressive manner in which they attacked a problem on which little data have been previously published. As they have mentioned, there are many applications where it would be rather unreasonable to use fatigue data of millions of stress cycles when the maximum life expectancy of a part may be only a few thousand cycles.

The authors have discussed somewhat the effect of cycling speeds on fatigue test results. It would be interesting to know at what speeds their testing was conducted and if they observed any effect of slow cycling speeds on the fatigue strength.

It would also be of great value if a short discussion could be presented on the appearance of the fracture surfaces of specimens failing at the various cycles tested.

MESSRS. H. J. GROVER[3] AND L. R. JACKSON[3] (*presented in written form*).—The authors have presented a very stimulating contribution to knowledge of fatigue properties important in design. Their new data on high-stress, short-lifetime behavior help fill in important gaps in available information.

However, we should like to ask some questions pertaining to interpretation of the test results:

1. In rotating-beam tests and in flexural tests, what allowance was made for plastic flow in evaluating stresses?
2. What was the estimated limit of error due to possible slight bending in reversed axial loading tests?

It appears that bending stresses, computed from the simple beam formula

$$S = Mc/I$$

without allowance for plastic flow, would be unduly high. On the other hand, tension stresses computed for axial-loading might be lower than those actually occurring (locally) if bending occurred due to very slight misalignment. These effects might explain part of the apparent discrepancy (see, for example, Fig. 7 in the paper) between results of fatigue tests of different kinds.

Considerations of local plastic flow in notched specimens are also of major importance in interpretation of results quoted in terms of nominal stress. This point is discussed in another paper[4] to be presented at this meeting.

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We agree with the authors that further work on the effects of a few cycles of high repeated stresses is needed. We should like to emphasize the importance of interpretation of experimental results in this region with respect to *plastic* as well as elastic behavior of metals.

MESSRS. M. H. WEISMAN AND M. H. KAPLAN (*authors' closure*).—The authors wish to express their appreciation to the discussers for their comments and contributions to the paper. Messrs. Jackson and Grover emphasize correctly the importance of plastic flow in high-stress fatigue tests. Following the original presentation of the paper, the results of the authors' flexural fatigue tests were recalculated to correct for the effect of plastic bending (see Table IV) and the *S-N* curves revised accordingly. The other flexural fatigue tests and the rotating-beam and reversed axial-loading fatigue tests were reported directly from the literature. No attempt was made to correct or revise the data except to recalculate the stresses to per cent of ultimate tensile strength for the purposes of this paper. Reference should be made to the original papers if specific information is desired concerning specimens or test procedures.

Mr. Piper properly interprets the comments in this paper to be a criticism of the practice of using for a design property of a given material the endurance strength determined by any one conventional fatigue test method. It is very important, as indicated in Mr. Piper's discussion, that the test method, testing speed, and stress range should simulate the design conditions in so far as possible. In particular, as noted by Mr. Zezula, there are many applications where it is unreasonable to use the endurance strength when it is known that the life expectancy is short.

The discussion in the paper pointed out that testing speeds up to 10,000 cpm. appeared to have no effect on the fatigue strengths of heat-treated aluminum alloys, such as 24S-T3 and 75S-T6. Tests now being conducted by other investigators will probably show the same result for quenched-and-tempered steels. Normalized and annealed steels and annealed aluminum and copper alloys have been reported to have lowered fatigue strength values when tested at speeds less than 100 cpm.

The tests described in this paper were all conducted at speeds of less than 50 cpm. The authors have not observed conclusive evidence of a speed effect in any of their other fatigue test work. It should be pointed out, however, that the great majority of such tests concerned individual parts tested to specific conditions. Thus the tests have been of such a nature that no indication of speed effect would normally be encountered.

The fractured surfaces of the axial load specimens reported in the paper are shown in Fig. 11. The fractures of the specimens loaded above the yield strength were of the cup-and-cone type similar to those normally obtained by the static tension test. Specimens tested at stresses less than the yield strength exhibited a shear type of fracture.

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EFFECTS OF GRINDING AND OTHER FINISHING PROCESSES ON THE FATIGUE STRENGTH OF HARDENED STEEL*

By L. P. Tarasov[1] and H. J. Grover[2]

Synopsis

This paper presents the results of an investigation of the effect of grinding and other finishing processes on the fatigue strength of a ball-bearing type steel, very similar to type 52100, at Rockwell hardness levels of C 45 and C 59. Grinding scratch direction was always parallel to the specimen length.

Reversed-bending tests of flat bars showed: (1) gentle grinding gave endurance limits as high as gentle grinding followed by careful polishing, (2) severe grinding reduced the endurance limits roughly 20 to 25 per cent, and (3) either abrasive tumbling or shot peening after grinding greatly increased the endurance limits.

Rotating-bending tests of round bars gave endurance limits about twice as high as reversed bending of flat bars of corresponding hardness. The endurance limits of round bars were affected relatively little by surfacing processes and the effects of a given process, if they were real, were not necessarily consistent for the two hardness levels.

In no case did gentle grinding prove detrimental to the fatigue properties.

Hardened steel parts generally have to be ground to satisfy dimensional requirements. Excessive heat resulting from improper grinding may crack, stress, or burn the surface, and these effects may easily have a detrimental influence on the service life of the part (**1**).[3] It is commonly accepted that surface cracks can be very harmful to the fatigue properties, but the picture is not at all clear as to how grinding severity may affect these properties in the absence of surface cracking.

Noll and Lipson (**2**), in classifying surface finishes for allowable working stresses, place in one category polished, ground, honed, lapped, and superfinished steel surfaces. They quote data by Horger (**3**), Hankins, Becker, and Mills (**4**), and Moore and Kommers (**5**) indicating little effect (0 to 11 per cent) of different finishes in this category on fatigue strength. However, the investigations mentioned are limited and include little in the way of controlled variation in grinding severity.

Mickel and Sommers (**6**) show an expected decrease in fatigue strength produced by actual grinding cracks. Staudinger (**7**) gives some interesting data on effects of grinding case-hardened and nitrided steel plates; he attributes some of the decrease in fatigue life to removal of surface layers having compressive stresses induced by the surface-hardening treatments. Boyer (**8**) reports results of rotating-bending fatigue tests on hardened type 52100 steel bars ground to various depths and then polished; although he concludes that the endurance limit is lowered by even the most careful grinding, there is reason to believe that the observed lowering is actually due to undetected burn effects, as pointed out by Tarasov in discussing Boyer's paper (**8**).

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Norton Co., Worcester, Mass.

[2] Battelle Memorial Institute, Columbus, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 686.

As there seems to be no other published information on the effects of grinding severity upon the fatigue strengths of steels of high hardness, a study of this was a major objective of the present investigation. In the course of the investigation, a number of surface finishes produced by other methods were also studied, both to furnish background and to answer some questions of practical interest.

The steel used throughout was a ball-bearing type steel having the following nominal composition:

Carbon, per cent	1.00
Chromium, per cent	1.40
Manganese, per cent	0.20
Vanadium, per cent	0.20
Silicon, per cent	0.25

This is essentially type 52100 steel with added vanadium. The steel was oil-quenched and tempered to give two hardnesses, Rockwell C 45 and C 59. The microstructure was tempered martensite throughout, containing fine, uniformly dispersed carbide particles. The hardness of the bars was uniform, being within 1.5 Rockwell C points of the nominal value.

The first part of the work was done on flat bars, which were tested in reversed bending. Flat bars were chosen as being much more amenable to *controlled* severe grinding than round bars. Two severities of grinding were obtained by using appropriate wheel grades and unit downfeeds. The "gentle" grinding represented, as nearly as possible, ideal grinding conditions; the "severe" grinding was intended to represent poor grinding practice but without producing any surface cracking. Other surfacing operations, which were superimposed on grinding, included hand polishing, electropolishing, shot peening, and abrasive tumbling.

Since most available data on the fatigue strength of high hardness steels are results of rotating-bending tests, the investigation was extended to the study of round bars. To avoid excessive distortion, it was necessary to grind with extreme gentleness, and only one set of bars was prepared under conditions expected to result in appreciable burn; however, this expectation was not fulfilled. The rest were ground gently and were either left in that condition or were subsequently hand polished, shot peened, abrasive tumbled, or grit blasted. In addition, some bars were ground and polished prior to heat treatment, for comparison with the bars whose surfaces were processed after heat treatment.

Both flat and round bars were ground in a longitudinal direction since, with the scratch direction parallel to that of the applied stress, any effect of surface profile on the fatigue data would be minimized. In this way, the physical effects of grinding were studied rather than those connected with the resulting surface geometry.

REVERSED-BENDING FATIGUE TESTS OF FLAT BARS

Specimen Preparation:

General Procedure.—After the bars had been machined from 0.250-in. stock to a thickness of 0.180 in. and approximately to the shape shown in Fig. 1, they were oil-quenched from 1550 F. and tempered 1 hr. at 400 or 890 F. to get the required hardness levels. Each bar was then ground wet to 0.160-in. thickness, this being done with extreme care to leave surfaces as flat and as stress-free as possible.

The elliptical contours were ground

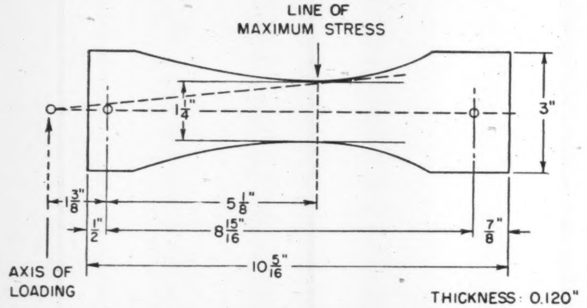

FIG. 1.—Flat-Bar Fatigue-Test Specimen.

NOTE.—The stress is maximum at the indicated line. It decreases less than 5 per cent for ½ in. either side of this line.

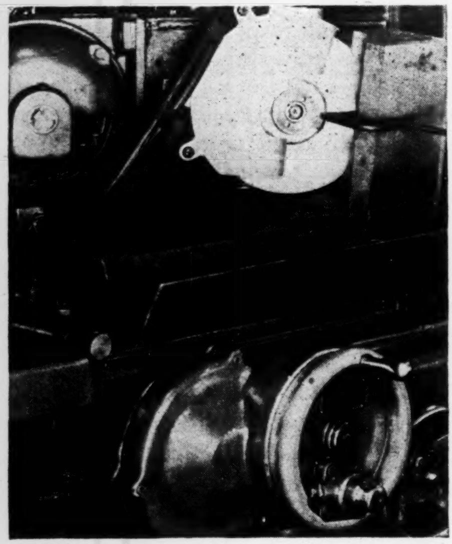

FIG. 2.—Setup for Grinding Elliptical Contours on Flat-Bar Specimens—General View.

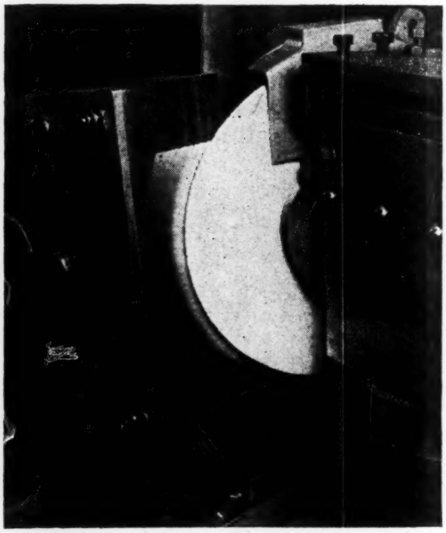

FIG. 3.—Setup for Grinding Elliptical Contours on Flat-Bar Specimens—Close-up Showing Mounting Jig.

dry on the setup shown in Figs. 2 and 3. Grinding was done with a 10-in. diameter, 32A46-F12VBEP wheel on a Norton No. 2 tool and cutter grinder. Infeed was adjusted to avoid burning; absence of burn was checked in preliminary experiments by etching the ground surface (9).

Next, each bar was ground lengthwise to its final thickness of 0.120 in. In all instances, this was done on a Norton 6 by 18-in. surface grinder under the following conditions:

Wheel size: 8-in. diameter, ½-in. width.
Wheel grade: Varied.
Unit crossfeed: 0.050 in.
Table speed: 65 ft. per min.
Wheel speed: 6,000 surface ft. per min.
Downfeed: Varied.

The desired grinding severity was obtained by varying wheel grade and downfeed.

Equal amounts were removed from the two sides of each bar. Curvature of the bar was measured (by determining arc height for the full length of bar) at each of three stages: before grinding, after one side was ground, and after both sides were ground. These determinations afforded indications of grinding severity and served as a control on the grinding operation.

In addition to measurements of changes in curvature, examinations were made on specimens of each group for surface smoothness, hardness and hardness variation, burn, and microstructure. Some of these examinations were made on sections cut after fatigue failure; however, results are quoted below to afford good descriptions of specimens tested.

Group C. Gently Ground Bars, Rockwell C59.—These were first ground gently with a soft wheel, 32A46-H8VBE, to an intermediate thickness of 0.140 in. Equal amounts were removed from the two sides, and the wheel was dressed sharp with a diamond prior to grinding each surface. Starting with this thickness, the wheel was dressed again and one surface was ground to give a thickness of 0.130 in.; unit downfeeds started at 0.0005 in. and finished with 4 downfeeds of 0.00025 in. and 6 of 0.0001 in. The wheel was dressed and the other surface was similarly ground to obtain a final thickness of 0.120 in.

The profilometer reading of the surface finish was about 20 microinches. Changes in curvature were so small (change in arc height for full length of bar averaging less than 0.001 in.) that residual surface stresses were estimated to be negligible. No traces of burn could be detected by etching metallographic taper sections of the bars after they had been broken in fatigue. It was concluded that the grinding conditions minimized any beneficial or detrimental effects upon the surface other than those possibly associated with a smoother finish.

Group D. Gently Ground and Hand-Polished Bars, Rockwell C59.—After being ground as described above, these were hand-polished on each surface. Polishing was done longitudinally with a piece of 240-grit Metalite cloth (Behr-Manning, aluminum oxide abrasive). Polishing for 30 sec., with spindle-oil lubricant, removed grinding scratches and gave a 10-microinch profilometer value. Change in arc height due to polishing one surface amounted to about 0.005 in. and corresponded to a low compressive stress (estimated at less than 30,000 psi.).

Group E. Severely Ground Bars, Rockwell C59.—These were ground with a very hard wheel, 32A46-L8VBE, using a 0.002-in. unit downfeed all the way. Each side was ground in the same manner to a final thickness of 0.120 in. Although grinding conditions were maintained as constant as possible, change in curvature varied greatly. About half of the bars ground were selected for testing; even so, the greater of the two measured changes in arc height (one for each surface) varied from 0.018 to 0.026 in. The generally high value was evidence of high residual surface tensile stress.

Profilometer readings were from 35 to 40 microinches. Surfaces were burned enough to be strongly overtempered, but there was no evidence of rehardening except in the bottoms of individual scratches. Burn was in the shape of streaks of uniform depth along the length of any one bar. Depth varied from bar to bar; extreme values were 0.001 and 0.004 in. For a representative bar, the drop in hardness due to burn (determined by a Tukon microhardness tester) was equivalent to about 4 points Rockwell C.

Group F. Severely Ground and Hand-Polished Bars, Rockwell C 59.—These were ground as in group E, polished for 2 min. with 80-grit Metalite cloth (to remove deep grinding scratches), and then polished as for group D.

Final profilometer values were about 10 microinches. Final surface stresses, judged by following changes in curvature, were still tensile although not so high as for group E. Burn depth due to grinding was like that for group E.

Group I. Gently Ground Bars, Rockwell C 45.—These were ground like those in group C except for a minor change: two instead of six final unit downfeeds of 0.0001 in. This produced a profilometer value of 10 microinches. No burn was detected by metallographic examination of taper sections. Changes in curvature were very small.

Group J. Gently Ground and Shot-Peened Bars, Rockwell C 45.—These were ground like the group I bars and then shot peened[4] as follows: 0.023 to 0.028-in. diameter chilled iron shot; 42 psi. air pressure for 10 min.; Almen A2 intensity 0.016 to 0.018 in. Profilometer reading was about 90 microinches.

Group K. Gently Ground and Electro-polished Bars, Rockwell C 45.—These were also ground the same as the group I bars and were then electro-polished. About 0.00075 in. was removed from each surface, and the resulting surface finish was about 7 microinches.

Group L. Severely Ground Bars, Rockwell C 45.—These were ground in the same manner as the harder group E bars, but resulting changes in curvature were smaller and much more reproducible. Changes in arc height varied between extreme values of 0.005 to 0.015 in., but most bars were in the range of 0.008 in. to 0.010 in. The surface finish was 35 to 40 microinches.

Although the ground surfaces were slightly discolored, no evidence of burn could be found in metallographic taper sections except for occasional rehardening or overtempering of the bottoms of individual scratches. Microhardness studies of two representative taper sections showed no evidence of surface softening; the only hardness change was an *increase* of 2 points Rockwell C because of work hardening by grinding.

Group M. Severely Ground and Shot-Peened Bars, Rockwell C 45.—These were ground like those of group L and shot peened like those of group J. The surface finish was about 100 microinches.

Group N. Severely Ground and Tumbled Bars, Rockwell C 45.—These were also ground like those of group L. They were then tumbled with 1000 lb. of Norton Alundum 2-T tumbling abrasive. In the $2\frac{1}{2}$-hr. roughing operation, 14 gal. of water and 1 oz. of Wyandotte No. 317 cleaner were used; 30 gal. of water and 3 oz. of cleaner were used in the 15-min. finishing operation. These conditions were selected as representative of commercial abrasive tumbling operations. The surface finish was about 15 microinches—much smoother than that from the shot peening.

Fatigue-Test Procedures:

Prior to testing, those bars which had sharp edges were gently polished with fine abrasive cloth to break the edges slightly. Figure 4 shows a specimen mounted in the bending-fatigue testing machine used. This machine loads the specimen as a cantilever beam. Deflections were measured by the dial indicator visible in the photograph, and maximum fiber stresses were calculated from these deflections by use of a calibration curve obtained for specimens of the same geometry and from material of the same elastic modulus as used in this investigation. Small inertial effects at the normal operating speed of about 1600 cpm. were included in the calibration.

As a further check, surface strains were measured dynamically by SR-4 gages on selected specimens. Each bar was clamped with special precautions to avoid bending or twisting due to clamping. Measurements indicated that, with the precautions used, applied stresses on top and bottom surfaces were equal to within about 500 psi., and stress was uniform across the width of the test section to within about this amount. It was estimated that errors in stress values due to loading techniques did not generally exceed about ±3 per cent.

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[4] These bars, as well as others to be described later, were shot peened by R. O. Collins of the Research Laboratories Division, General Motors Corp.

Fatigue failure, which always fractured the test specimen, stopped the machine within about 1000 cycles.

Fatigue-Test Results:

Results of fatigue tests on the flat bars are shown in the *S-N* curves of Figs. 5 and 6. Points with arrows designate specimens which did not fail or, in two instances, specimens which did not fail in the test section due to premature failure in grips. For all others, failure was essentially in the region of maximum stress.

Fatigue fractures were conventional in appearance and usually showed a crescent-shaped smooth region centered about the origin of the crack leading to failure. (More than one crescent appeared in a number of instances—and multiple cracks were sometimes noted.) Failure always started in the highly-stressed outer fibers (either at top or bottom surface). Although account was kept of location of failure with respect to which surface showed most change in curvature during grinding, no correlation appeared. Again, while failure often started at a corner, many failures also started near the center. In general,

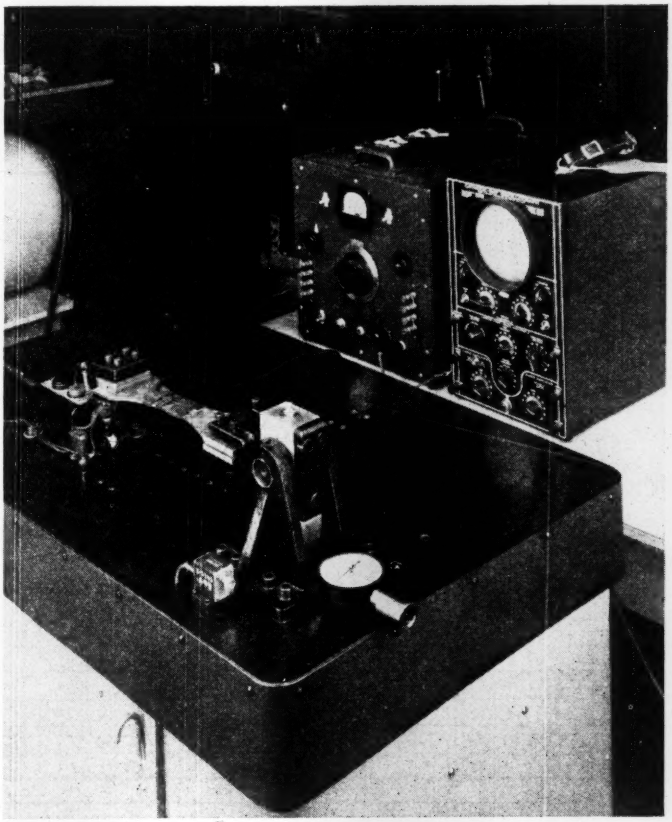

FIG. 4.—Flat-Bar Specimen in Fatigue-Testing Machine.

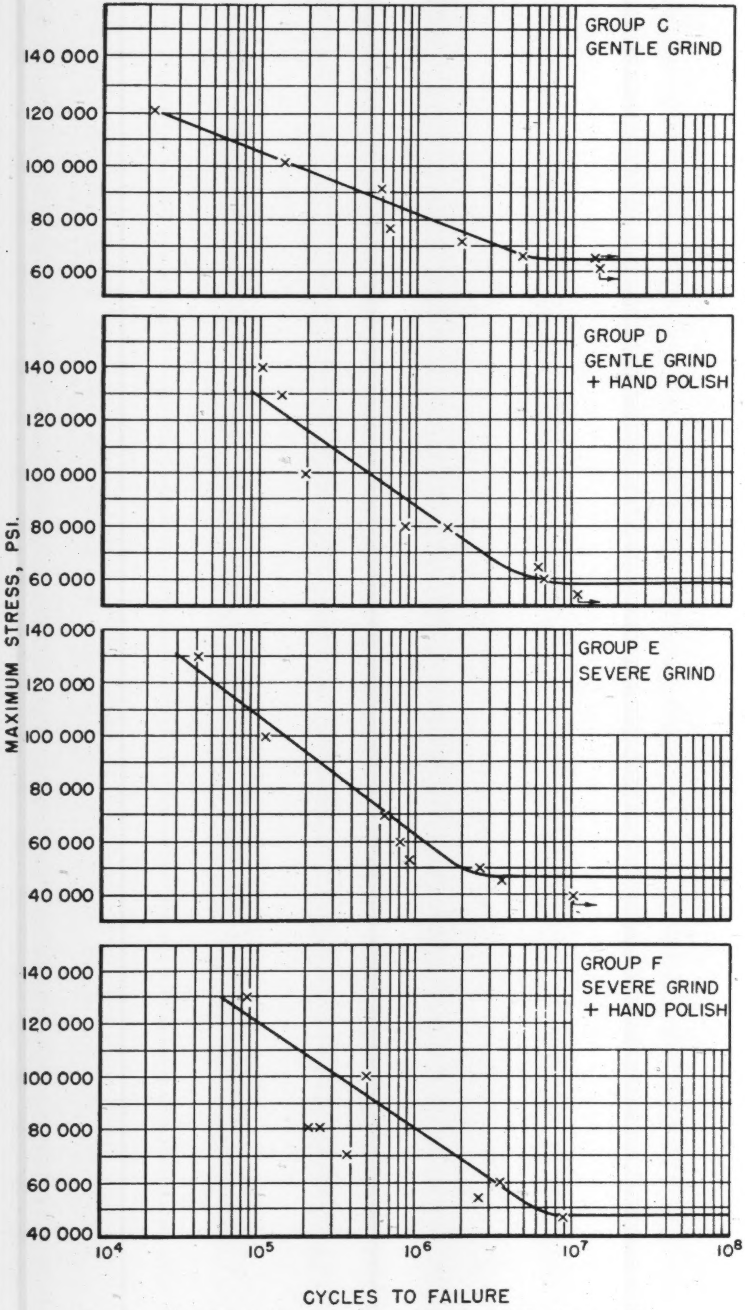

FIG. 5.—*S-N* Curves for Flat Bars of Rockwell Hardness C59.

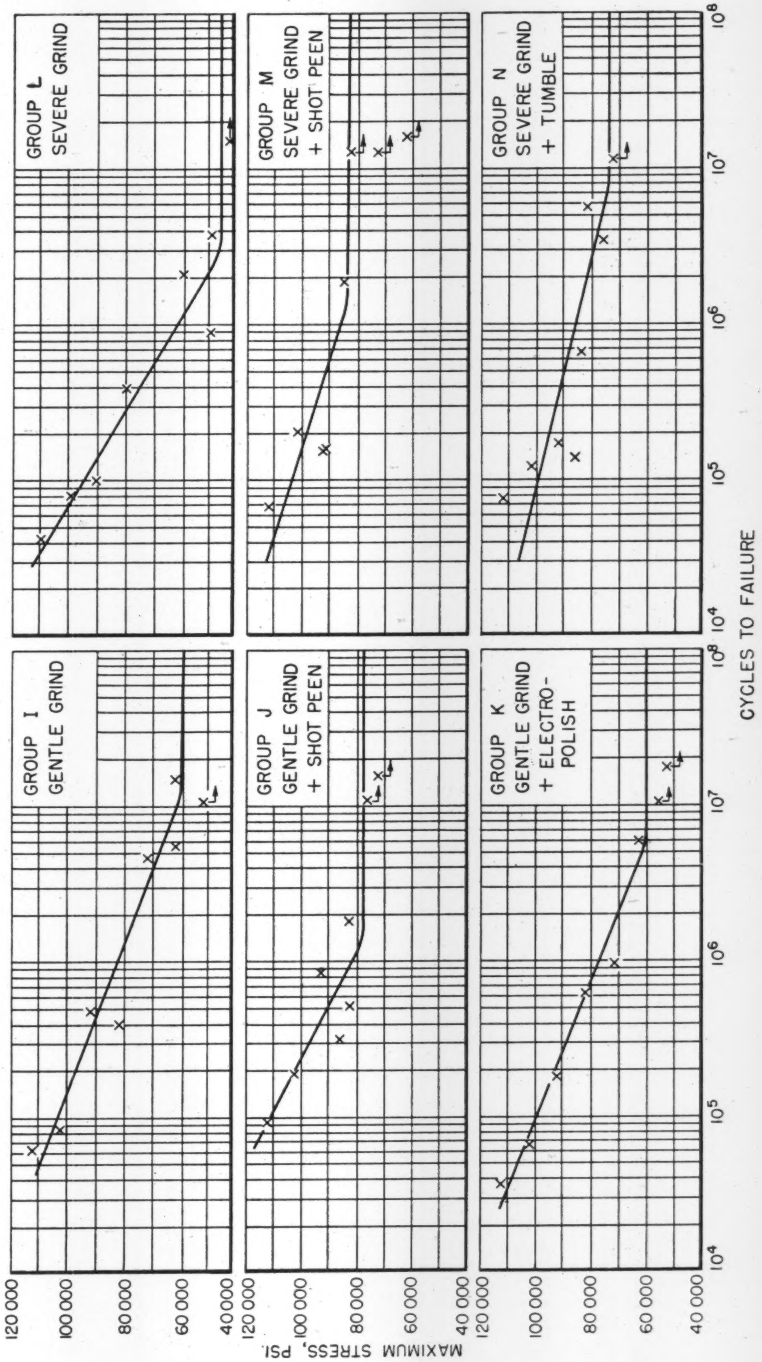

FIG. 6.—*S-N* Curves for Flat Bars of Rockwell Hardness C45.

neither location nor appearance of failure showed any correlation with unusually long or short specimen lifetime.

ROTATING-BENDING FATIGUE TESTS OF ROUND BARS

Specimen Preparation:

General Procedure.—Figure 7 shows the round-bar fatigue test specimen. All bars, whether in the annealed or hardened condition, were ground wet on a Norton No. 2 tool and cutter grinder with a 7-in. diameter, 1-in. wide 32A60-G8VBE wheel running at 5000 surface ft. per min. The setup is shown in Fig. 8. The initial diameter of the minimum section was 0.250 in. while the final diameter was 0.230 in. The grinding fluid was a commercial soluble oil diluted 40 to 1 with water. The bar, rotating at 180 rpm., was traversed at a moderate rate across the wheel, which was fed down 0.0005 in. at the end of each crossfeed until the bar was only 0.001 in. oversize. At this point, the unit downfeed was reduced to about 0.0001 in. The grinding scratches were almost exactly longitudinal since the wheel and work surface speeds were in the ratio of approximately 500 to 1. The grinding conditions were extremely gentle so as to keep the bars as straight as possible and to avoid burning the hardened bars. Special grinding conditions for the dry-ground bars are described later. For each of the following sections, the first group of bars listed was heat treated to Rockwell C 45, and the second one to Rockwell C 59.

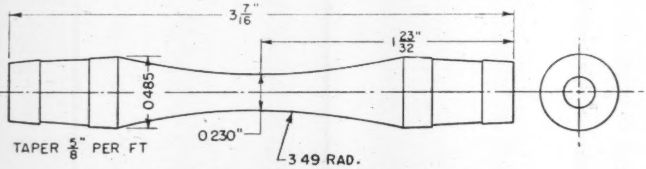

FIG. 7.—Round-Bar Fatigue-Test Specimen.

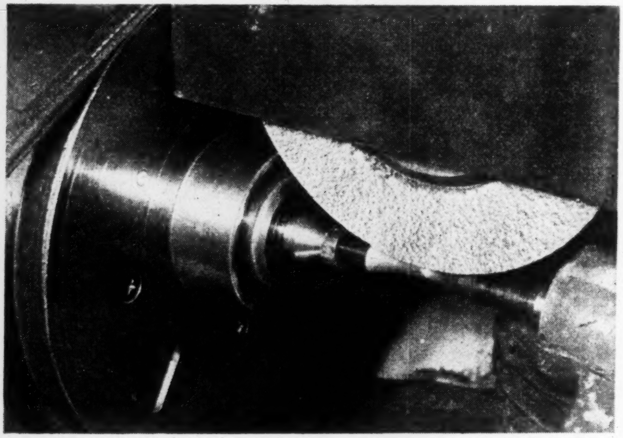

FIG. 8.—Setup for Grinding Round Bars.

Groups MA and MG. Bars Heat Treated After Gentle Grinding.—These bars were ground in the annealed state, as just described. The longitudinal grinding scratches were removed by polishing with a series of abrasive papers, the bar being rapidly rotated in a drill press. The resulting circumferential scratches were removed by hand polishing longitudinally with 240-grit abrasive paper. The bars were then copper plated in cyanide solution to prevent changes in chemical composition of the surface during subsequent heat treatment. This consisted of oil quenching from 1550 F., followed by tempering the MA group for 1 hr. at 850 F. to Rockwell C 45 and tempering the MG group for 1 hr. at 400 F. to Rockwell C 59. After the copper plating was removed by treatment in a sodium cyanide solution, the bars were polished very lightly in the longitudinal direction with No. 000 metallographic emery paper impregnated with a suspension of 6-μ diamond dust in olive oil. Bars of both these groups gave profilometer readings averaging about 5 microinches.

After the fatigue tests were completed, careful examination of a nickel-plated taper section showed that the microstructure and microhardness were exactly the same at the surface as in the interior. The carbon content at the surface obtained from analysis of a layer 0.0015 in. thick, taken in the vicinity of the minimum diameter, was the same as that of the steel below the surface. Thus, the plating technique was fully effective in eliminating any surface changes in chemical composition which might affect the fatigue data.

Groups ME and MK, Gently Ground Bars.—These were ground gently in the hardened condition. The heat treatment was the same as for the MA and MG bars, respectively, but the plating was, of course, omitted since any surface changes in chemical composition were eliminated in the early stages of grinding. The grinding scratches were rather deep because of the nature of the grinding contact. Examination of a metallographic taper section showed that the surface was not burned in the least and that its microhardness was exactly the same as that of the interior. Profilometer values were about 20 microinches for ME bars and about 15 microinches for MK bars.

Groups MD and MJ, Gently Ground and Hand-Polished Bars.—These were heat treated and ground like the bars immediately above. The longitudinal grinding scratches were then removed by hand polishing with a series of aluminum oxide abrasive papers, ranging from 80 to 240 grit, using kerosine for a lubricant. The bar was alternately polished circumferentially while rotating in a drill press and then longitudinally while stationary. The final scratches, produced with the 240-grit paper, were longitudinal. Profilometer readings were 7 microinches for MD and 5 microinches for MJ.

Groups MB and MH, Gently Ground and Tumbled.—The bars were heat treated and ground like those in groups ME and MK. They were abrasive tumbled like the flat bars in group N except that the roughing operation was made an hour longer in order to eliminate more completely the deep grinding scratches. Profilometer values for both groups were about 5 microinches.

Groups MC and MI, Gently Ground and Shot Peened.—After being processed like the ME and MK bars, these were shot peened like the flat bars in groups J and M except that the air pressure was decreased to 37 psi. and the time to 2 min. to compensate for the difference in size between the flat and round bars and also for the use of another peening machine having different shot flow rates. The peening intensity, as measured on the Almen strip, was thus kept the same for the round bars as for the flat bars; however, this did not insure the same surface effect on the two different types

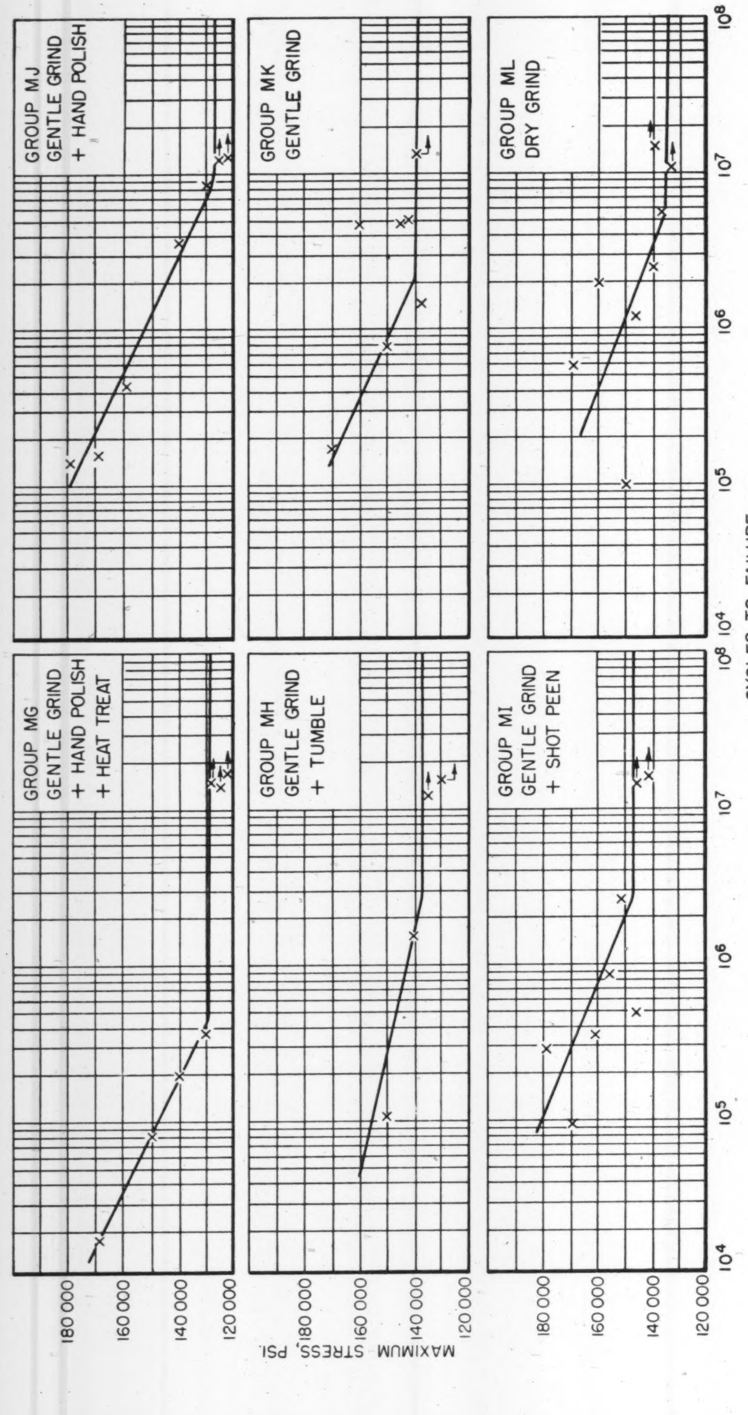

FIG. 9.—*S-N* Curves for Round Bars of Rockwell Hardness C59.

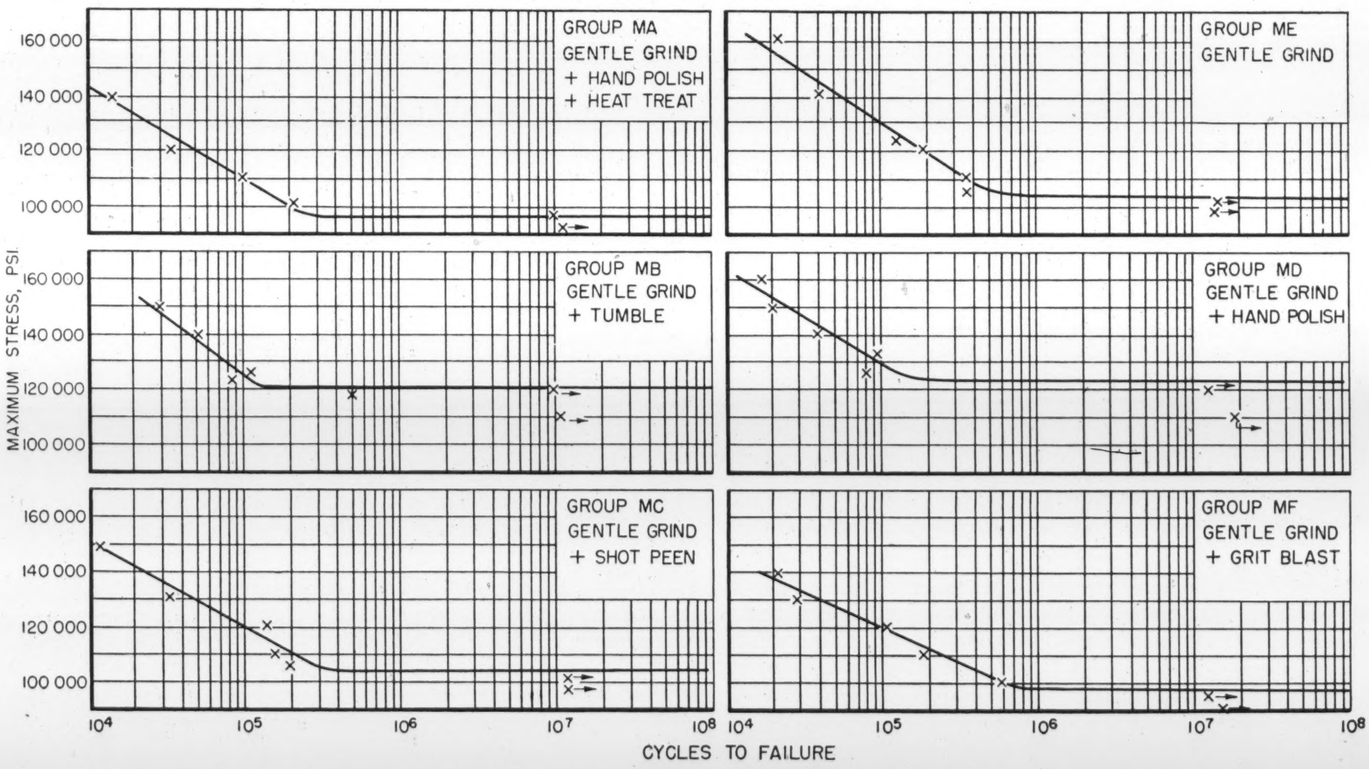

FIG. 10.—*S-N* Curves for Round Bars of Rockwell Hardness C45.

of bars. Profilometer readings were about 45 microinches for bars of group MC and about 35 microinches for bars of group MI.

Group MF. Gently Ground and Grit Blasted.—These bars, heat treated to Rockwell C 45, were processed like the ME bars and were then blasted for 25 to 30 sec. with No. 60 steel grit while being rotated at about 120 rpm. The blast pressure was about 80 psi., and the nozzle was about 6 in. from the work. MF bars had a rather rough surface with a profilometer value of about 170 microinches.

Group ML. Dry-Ground Bars.—These bars, heat treated to Rockwell C 59, were ground in different manner than any of the above in an attempt to obtain a severely ground surface. They were first ground wet exactly the same as the gently ground bars until the minimum diameter became 0.237 in. The grinding fluid was then turned off and the diameter was reduced to 0.233 in. in unit downfeeds of 0.0005 in. Even though the wheel was extremely soft, enough heat was built up in the bar to warp it appreciably. Wet grinding was then resumed, the unit downfeed being cut to about 0.0001 in. The warpage was just eliminated by the time the final diameter of 0.230 in. was reached. Nearly all the stock was removed from one side of the bar so that probably all of the dry-ground zone in that portion was eliminated from the finished specimen. The remaining portion of the surface, from which very little, if anything, was removed in the final gentle grinding operation, was more or less in the condition in which it was left by the dry-grinding operation. It is highly probable that the surface condition of these bars varied much more from one bar to another than it did for any of the other groups of round bars. Although it had been expected that the dry-grinding operation would burn the surface, no evidence of burn could be found metallographically.

Fatigue-Test Procedure:

Round bar specimens were tested in conventional R. R. Moore-type fatigue testing machines at speeds of about 10,000 rpm. The minimum diameter of each bar was measured on a calibrated shadow graph (since micrometers might have marred the surface), and the stress value was computed on the basis of the individually measured section diameter. Known sources of error in loading were not considered to influence computed stresses by more than 1.5 per cent.

Fatigue-Test Results:

Results of the rotating-bending fatigue tests on round bars are shown in the *S-N* curves of Figs. 9 and 10. Points with arrows represent specimens removed before failure. Fatigue fractures were quite conventional in appearance; nothing unusual enough to warrant comment was noted.

DISCUSSION OF RESULTS

Evaluation of Endurance Limits:

The following discussion is based on endurance limits estimated from the *S-N* curves in Figs. 5, 6, 9, and 10. Examination of these figures indicates scatter of points from the faired curves drawn. In many cases, this scatter is greater than estimated experimental error in testing (see sections on test procedures). Even when scatter appears small, it is unsafe to regard an *S-N* curve, based upon a limited number of specimens, as giving an accurate measure of fatigue strength of a material; see, for example, the report of Ransom and Mehl (10) on statistical variations in fatigue testing.

A later section of this discussion contains some speculative remarks on the possibility of scatter being associated with variable surface preparation. However, even in comparing endurance limits observed on bars differently surfaced, it is necessary to keep in mind the fact that the endurance limits were estimated from tests of limited numbers of specimens, and the precision of the estimated values in unknown. For definiteness, it will be considered that a difference of 10 per cent or less between any two endurance limits is insignificant.[5]

Evaluation of Data for Flat Bars:

Table I shows the effect of grinding severity on endurance limits of the flat bars. The severe grinding decreased the endurance limit about 25 per cent below values for gently ground bars.

TABLE I.—EFFECT OF GRINDING SEVERITY (FLAT BARS).

Group	Rockwell Hardness, "C" Scale	Grinding	Additional Processing	Endurance Limit, psi.
C........	59	Gentle	None	65 000
E........	59	Severe	None	45 000
D........	59	Gentle	Hand polishing	59 000
F........	59	Severe	Hand polishing	47 000
I........	45	Gentle	None	60 000
L........	45	Severe	None	45 000
K........	45	Gentle	Electrolytic polishing	60 000

It is also clear, from Table I, that neither hand nor electrolytic polishing after grinding significantly affected endurance limits. It is possible that the polishing did not remove enough metal to have maximum influence. However, as previously noted, polishing did decrease surface roughness (in the case of severely ground bars from 35–40 microinches). It may be concluded that smoothness alone is not an adequate criterion of surface conditions influencing fatigue endurance limits.

Since gentle grinding left no significant residual stress (on the basis of curvature measurements) and since further smoothing of the surface did not increase fatigue strength, results for the gently ground bars appear representative of the bending fatigue strength of the material in the flat test specimens used. Had the scratch direction been perpendicular to the direction of applied stress instead of parallel, the fatigue data might have been somewhat lower because of the notch effect of such scratches.

TABLE II.—EFFECTS OF SHOT PEENING AND OF ABRASIVE TUMBLING (FLAT BARS).

Group	Rockwell, Hardness, "C" Scale	Grinding	Additional Processing	Endurance Limit, psi.
I........	45	Gentle	None	60 000
J........	45	Gentle	Shot peening	78 000
L........	45	Severe	None	45 000
M........	45	Severe	Shot peening	83 000
N........	45	Severe	Tumbling	73 000

Table II summarizes effects of shot peening and of tumbling. The apparently greater endurance limit for the severely ground and shot-peened bars (M) than for the gently ground and shot-peened ones (J) has no significance in view of the uncertainty in these values. Both shot peening and abrasive tumbling greatly increased the observed endurance limit. Damage done by severe grinding was not only eliminated, so far as endurance limit was concerned, but the endurance limit of severely ground bars was raised, in these particular instances, above the value for bars left in the gently ground condition. This should not be misinterpreted as advocacy of poor grinding practice, which may lead to difficulties not capable of being corrected by shot peening or tumbling.

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[5] This value is entirely arbitrary and not to be considered as an estimate of precision of absolute values. It may be pointed out, however, that comparing endurance limits of bars of the same steel, very carefully processed in the same laboratory and tested upon the same machine may be justifiable to a higher precision than comparing results of tests on different lots of material. In general, groups of bars to be compared with each other in this discussion were prepared at the same time from the same lot of material by the same operator and were tested in one machine by one operator. (This does not apply to comparison of results of tests of round bars with results of tests of flat bars.)

Similar increases of fatigue strength have been previously reported for shot peening, but this is thought to be the first evidence that abrasive tumbling can have a beneficial effect on fatigue strength. While tumbling did not, in this case, increase the endurance limit as much as shot peening, it should be remarked that there was no background available as to optimum conditions of tumbling to improve fatigue resistance. Whether even greater benefits can be obtained from tumbling remains to be investigated.

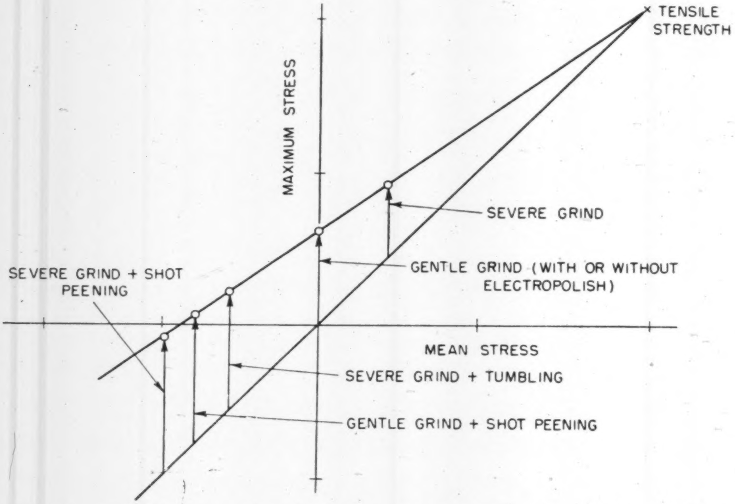
TENSILE STRENGTH
MAXIMUM STRESS
SEVERE GRIND
GENTLE GRIND (WITH OR WITHOUT ELECTROPOLISH)
SEVERE GRIND + SHOT PEENING
MEAN STRESS
SEVERE GRIND + TUMBLING
GENTLE GRIND + SHOT PEENING

FIG. 11.—Goodman-Type Diagram Illustrating Possible Interpretation of Effect of Residual Stresses on Observed Stress Range at Endurance Limit.

Most of the effects noted for the flat bars can be at least partly explained in terms of residual surface stresses. This point of view is illustrated in Fig. 11, on the basis of an assumed Goodman-type diagram. The residual surface tensile stress due to severe grinding may be considered to increase the mean stress of the loading cycle above zero and thereby decrease the range of stress observed to cause fatigue failure. Similarly, residual surface compression stresses (such as due to the shot peening or, presumably, to the tumbling) decrease the mean stress below zero and thereby increase the range of stress required to cause fatigue failure. Such a mechanism, in contrast to a real increase in the intrinsic reversed-stress endurance limit of the material, probably plays an important rôle in causing the nominal increased endurance limits reported. However, the diagram shown in Fig. 11 must be considered only schematic since (1) the actual shape of the Goodman diagram for this steel is unknown, (2) residual stress values were not measured other than qualitatively by change in curvature, and (3) other factors (surface smoothness, shape and size effects, stress gradients, etc.) should not be lightly dismissed.

Evaluation of Data for Round Bars:

In contrast to the flat bars, the round

bars showed relatively little effect of the various surfacing processes upon endurance limit. However, the round bars did have much higher endurance limits than the flat bars.

Table III shows endurance limits for bars ground either before or after heat treatment. The bars having a hardness of Rockwell C 59 ground before heat treatment (MG) had a lower endurance limit than those ground after heat treatment (MK); the same may have been true of the softer bars, although the difference in this case was too small to be reliable. No reason could be found for this behavior. The ground and heat-treated bars were uniform in carbon content, microstructure, and microhardness out to the surface; they were expected to have the same endurance limits as the bars gently ground after heat treatment.

TABLE III.—EFFECTS OF HEAT TREATING BEFORE AND AFTER GRINDING (ROUND BARS).

Processing		Rockwell C 45		Rockwell C 59	
Preliminary	Additional	Group	Endurance Limit, psi.	Group	Endurance Limit, psi.
Gentle grinding	Polishing and heat treatment	MA	94 000	MG	128 000
Heat treatment	Gentle grinding	ME	102 000	MK	140 000
Heat treatment	Dry grinding	...	....	ML	136 000

The dry grinding was intended to be severe, in contrast to the very gentle wet grinding; actually, no signs of burn could be detected upon subsequent examination of dry-ground bars. The endurance limit was the same as for those ground wet in the heat-treated condition. It should be noted that all values for groups of equal hardness in Table III are within a ±5 per cent scatter band.

The endurance limits for other groups of round bars are shown in Table IV. Again, values for groups of equal hardness lie mostly within a scatter band of ±5 per cent so that differences may not be significant. Hand polishing appeared to lower the endurance limit for the bars with a hardness of Rockwell C 59 and to raise the endurance limit for the bars having a hardness of Rockwell C 45. No reason for this behavior was found; the only explanation that can be offered is that some unknown factor associated with the polishing or with the material varied sufficiently to affect the two sets of bars so differently.

TABLE IV.—EFFECTS OF MECHANICAL PROCESSING FOLLOWING GENTLE GRINDING (ROUND BARS).

Additional Processing	Rockwell C 45		Rockwell C 59	
	Group	Endurance Limit, psi.	Group	Endurance Limit, psi.
None	ME	102 000	MK	140 000
Hand Polishing	MD	122 000	MJ	127 000
Shot Peening	MC	102 000	MI	147 000
Abrasive Tumbling	MB	120 000	MH	137 000
Grit Blasting	MF	97 000	..	..

Shot peening did not have any significant effect on the endurance limit. Tumbling raised the endurance limit slightly for the bars with a hardness of Rockwell C 45 but not for the others. Only one set of bars was grit blasted and their endurance limit was not significantly influenced.

While the processing of round bars and flat bars could not be considered identical for all types of surfacing, this difference does not simply explain the widely different magnitudes of endurance limits. It is probable that a part, at least, of the difference was due to the difference in shape and size of the test bars (flat *versus* round) and the difference in load-

TABLE V.—SOME COMPARISONS OF ENDURANCE LIMITS FOR ROUND AND FLAT STEEL BARS.

Source[a]	Tensile Strength, psi.	Specimen Dimension, in.			Endurance Limits, psi.		Ratio, Flat/Round
		Round Bar Diameter	Flat Bar				
			Width	Thickness	Round	Flat	
1......	75 000	0.30	0.40	0.40	49 000	46 000	0.94
2......	129 000	0.19	1.00	0.19	73 000	62 000	0.85
1......	150 000	0.30	0.32	0.32	80 000	80 000	1.00
2......	150 000	0.19	1.00	0.19	85 000	72 000	0.85
3......	192 000	0.23	1.25	0.125	90 000[b]	72 000	0.80
2......	206 000	0.19	1.00	0.19	115 000	74 000	0.64
4......	210 000[c]	0.23	1.25	0.12	102 000	60 000	0.59
4......	320 000[c]	0.23	1.25	0.12	140 000	65 000	0.45

[a] Sources: 1.—from Discussion by Dolan (**13**).
2.—from paper by Fuller and Oberg (**13**).
3.—from paper by Grover, Bennett, and Foley (**14**).
4.—present paper: groups ME, I, MK, and C.

[b] Value adjusted for slight difference in tensile strengths of bars.

[c] Estimated from Rockwell hardness, *Metals Handbook*, p. 99 (1948).

ing (reversed plane bending *versus* rotating bending).

H. F. Moore (**11**) and others have shown that rotating-bending fatigue strength tends to decrease with increasing diameter of specimen—particularly in the range from $\frac{1}{8}$ to $\frac{1}{2}$ in. One interpretation has been size effect: that larger specimens have larger volumes of highly stressed material and that such larger volumes have, statistically, a greater number of weak spots. Another suggestion has been that stress gradient (paper by Peterson (**12**)) is important in determining fatigue strength and larger diameter specimens have, for a given outer fiber stress, lower stress gradient than smaller specimens. The flat bars used in this investigation had about twice the volume of critically stressed material as the round bars; the flat bars also had about twice as high a stress gradient as the round bars. In addition, the flat bars had sharp corners in contrast to the round bars; however, failure did not always occur at corners. Testing speeds were also different for the two sets of bars; however, speed effects in this range usually have been considered negligible for steels at room temperature in noncorrosive environment.

Whatever the cause or causes of the difference in results of tests on flat bars and on round bars, this difference is not out of line with some other published results. Table V shows some comparisons of endurance limits determined on flat bars in plane bending and on round bars

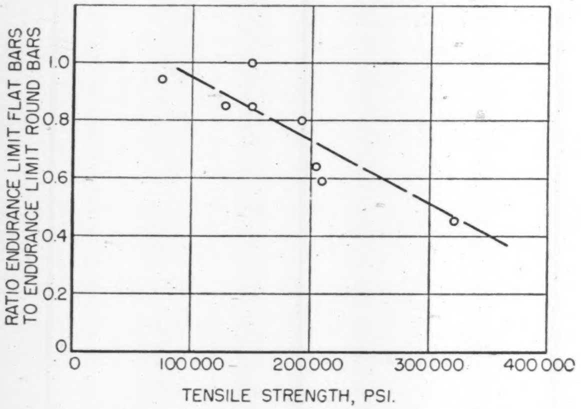

FIG. 12.—Ratios of Flat-Bar Endurance Limit to Round-Bar Endurance Limit for Steels of Increasing Hardness.

in rotating bending. Only values for gently ground bars have been included from this investigation, since it seems likely that only these have similar surfaces in the two shapes; for details of surfacing specimens in previously reported studies, the references cited should be consulted. Figure 12 shows the ratio of flat-bar endurance limit to round-bar endurance limit plotted against ultimate tensile strength. A trend for this shape factor to decrease with increasing hardness of the steel is apparent. Results of the present investigation are in line with this trend.

Scatter of Points on S-N Curves:

Since extreme care was used in surfacing the test specimens in this investigation, it appeared interesting to observe to what extent scatter of points might correlate with surface conditions of individual specimens. In view of the limited number of specimens for any curve, such observations must be considered wholly speculative.

For the data on flat bars, scatter could not be correlated with changes in curvature during processing, with burn depth, or with small variations in Rockwell hardness. However, there appears a possibility that the degree of scatter was influenced by the manner of processing. Scatter was low for gently ground bars (C and I) and for the electro polished ones (K). It was also low for the severely ground bars with hardness of Rockwell C 59 (E) but medium for the severely ground group with hardness of Rockwell C 45 (L). Hand polishing increased the scatter both after gentle grinding (D) and after severe grinding (F). Shot peening (J and M) resulted in medium scatter, and tumbling (N) in high scatter. It thus appears that the gentle grinding, whether or not followed by electrolytic polishing, resulted in the most uniform test specimens and that the hand polishing destroyed some of the uniformity. This seems reasonable since the gentle grinding employed might be expected to be more reproducible in its effect on the surface than polishing by hand with abrasive cloth. Operations like severe grinding, shot peening, and tumbling might also be expected to result in some nonuniformities in surface.

A similar analysis of the scatter of data on the round bars failed to corroborate the trends shown by tests on flat bars. This apparent lack of agreement may be partly due to the appreciable roughness of the round bars in the gently ground condition which was not completely eliminated in subsequent processing; occasional scratches in an otherwise smooth surface are not recorded by the profilometer. Moreover, most surface treatments of round bars were not so well documented (for example, by observation of change of curvature) as treatments of flat bars; therefore, unnoticed variations in surfacing may have occurred. It is worth noting that in the two groups (MA and MG) of bars heat treated after grinding and polishing in the annealed state, the polishing was much more effective in removing the grinding scratches, and the scatter was low.

These speculations on possible origins of scatter in S-N curves suggest that further work along the lines of careful study of surface conditions might be profitable in uncovering some of the variables contributing to scatter of results of fatigue tests.

CONCLUSIONS

1. Very careful longitudinal grinding of hardened steel fatigue test bars, whether flat or round, did not have any adverse effect on their fatigue strength. Contrary reports, published and otherwise, are attributed to unrecognized

defects resulting from faulty grinding practice.

2. Severe grinding in a longitudinal direction appreciably lowered the reversed-bending endurance limits of flat bars. The decrease was roughly 20 to 25 per cent for the particularly severe grinding conditions used—a much harder wheel than proper, and a 0.002-in. unit downfeed. The harder bars (Rockwell C 59) so ground were badly overtempered, while the medium hard (Rockwell C 45) bars were not detectably burned; however, bars of both sets were severely stressed in tension. The grinding was not abusive enough to reharden the surface or to cause surface cracking of the steel used, or the drop in endurance limit might have been correspondingly greater.

3. Shot peening and abrasive tumbling both increased the endurance limits observed for the flat bars. The endurance limit of severely ground flat bars was increased by these processes to values above that for bars left in the gently ground condition. This does not imply that defects resulting from poor grinding practice can necessarily be corrected in this manner.

4. Reversed-bending endurance limits of flat bars were lower, roughly by a factor of two, than rotating-bending endurance limits of round bars. The ratio of flat to round-bar endurance limits of steel apparently decreases with increasing tensile strength.

5. Effects of surface processing on the round-bar endurance limits were less pronounced (and less obviously explainable) than in the case of flat bars.

6. In view of items 4 and 5 above, results of rotating-bending tests of round bars do not afford adequate evaluation of surfacing processes for all design purposes.

7. In the preparation of fatigue test specimens, attention should be paid not only to the geometrical nature of the finish but also to possible physical changes in the surface layers during preparation.

Acknowledgments:

The authors express their appreciation to the numerous people in the Norton and Battelle organizations whose efforts at various stages of the work made this paper possible. Particular mention should be made of the work of Mr. C. O. Lundberg, of Norton Co. Research Laboratories, in supervising the preparation of all the test bars, in grinding and polishing them properly, and in assisting, together with Mr. H. L. Ericson, in the determination of their surface condition. Mr. J. M. Berry, of Battelle, directed much of the fatigue testing and assisted in the examination of failed specimens. The authors also acknowledge helpful suggestions from Mr. L. R. Jackson, of Battelle Memorial Institute, in planning the investigation and in interpreting the results obtained.

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DISCUSSION

MR. J. O. ALMEN[1] (*presented in written form*).—It is a pleasure to find that the Norton Co., through Messrs. Tarasov and Grover, is earnestly seeking for the truth in regard to the effects of grinding upon the fatigue strength of ground surfaces. Although the evidence will necessarily confirm my oft-repeated contentions that actual and extensive loss of fatigue strength is inflicted on ground machine parts, the publication of these facts will go far to reduce abusive grinding room practices.

It is hoped that future papers will include actual measured residual stresses in the surfaces of fatigue specimens, since the data will not become intelligible until the actual stress is known. The present paper misinterprets many important findings because of unwarranted assumptions. Until measurements are made of the residual stress magnitude at the extreme surface of the specimen the conclusions regarding the failure stress are little more than guesses. The statement on page 669 of the paper that "... the physical effects of grinding were studied rather than those connected with the resulting surface geometry," would be justified if the actual stress had been measured.

FATIGUE STRENGTH GAIN OR LOSS EXPLAINED

The reasons for the gain or loss of fatigue strength that results from the various surface finishing operations that are discussed by Tarasov and Grover have been presented in a number of publications[2, 3, 4, 5] that apparently have escaped the attention or the serious consideration of the authors. These publications advance the concepts and present supporting data which, when the actual stresses are known will be further confirmed by the Tarasov and Grover paper, that

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[1] Executive Consultant, Research Laboratory Division, General Motors Corp., Detroit, Mich.

[2] J. O. Almen, "Peened Surfaces Improve Endurance of Machine Parts," *Metal Progress*, February, 1943, pp. 209–215.

[3] J. O. Almen, "Shot Blasting to Increase Fatigue Resistance," *Transactions*, Soc. Automotive Engrs., Vol. 51, No. 7, July, 1943, pp. 248–268.

[4] J. O. Almen, "Shot Peening," *Kent's Mechanical Engineers Handbook*, Twelfth Edition, Design and Production, Wiley Handbook Series, pp. 20–40 to 20–49, John Wiley and Sons Inc., New York, N. Y. (1950).

[5] J. O. Almen, "Mechanical Wear," Am. Soc. Metals, pp. 229–288 (1950).

1. The fatigue strength of surfaces is much lower than the fatigue strength of sound sub-surface metal.
2. Fatigue failures are tensile failures.
3. The yield strength of metals under repeated loads is much lower than the yield strength as measured by static tests.

Although normal commercial grinding may sometimes increase the fatigue strength of severely "notched" specimens by reducing the magnitude of local tensile stress raisers, it decreases the fatigue strength of surfaces that are reasonably smooth. Since commercial grinding induces residual tensile stress in a thin surface layer, the fatigue strength of smooth specimens is reduced by an amount approaching the magnitude of the *retained* residual tensile stress that is induced by grinding.

Commerical grinding induces residual tensile stress in the ground surface because the metal immediately under the wheel at any instant is severely heated by the local work of rapidly cutting and tearing metal particles from the surface. The hot metal seeks to expand thermally, but being restrained by adjacent cold metal, the hot area becomes compressively stressed. When this stress exceeds the transient yield strength of the metal at the instantaneous temperature of the hot spot, compressive plastic yielding occurs. As the grinding wheel advances, the plastically deformed metal cools and contracts, whereupon residual tensile stress is developed in magnitude and depth proportional to the extent of plastic compressive yielding.

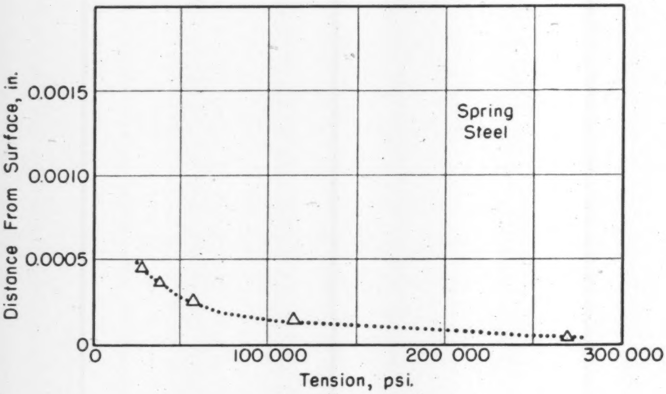
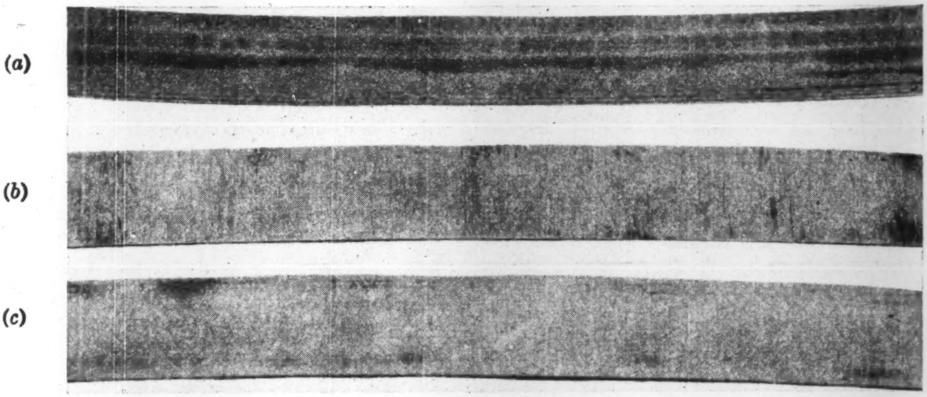


FIG. 13.—Residual Stress Caused by Grinding.

GRINDING STRESS MEASURED

Residual stress measurements by the Research Laboratories Division of the General Motors Corp. indicate that, depending upon the manner of grinding, tensile stress may approach the ultimate strength of hard steel without cracking as is shown in Fig. 13 which is reproduced from an earlier publication.[3] This chart records residual tensile stress in spring steel of approximately 275,000 psi.

When metal is removed by a cutting tool at a rate that does not develop appreciable heat, the residual stress that is induced is found to be compressive. This is presumably the result of plastic "smearing" of the surface metal by the cutting tool. Thus honing and filing induced residual compressive stress as is shown in Fig. 14(*b*) and (*c*) by the fact that these specimens are curved convex on the honed and filed surfaces in contrast to the concave curvature of the ground specimen (Fig. 14(*a*)). Normal grinding also "smears" the surface metal, and residual compressive stress would result except for the transient heat and consequent compressive plastic yielding that occurs. The local heat of grinding and the resulting residual tensile stress is reduced as the local work of cutting away metal particles is reduced. A grinding wheel having sharp cutting edges generates less heat than a wheel having dull cutting edges. Sharper cutting edges are assured when the wheel is frequently dressed. Also a wheel having a weak binding material will be sharper than a wheel having a strong binding material because dull granules will be easily broken away as they become dull, whereas strong binders retain dull granules which increase local pressure and frictional heat. Local heat also decreases as the depth of the cut is decreased.



(*a*) Grinding (Tension). (*b*) Filing (Compression). (*c*) Honing (Compression).
FIG. 14.—Curvature of Specimens Shows Stress Induced in Surface by Various Treatments.

GRINDING STRESS REDUCED

By the use of very soft wheels and extremely shallow cuts the Research Laboratories Division of the General Motors Corp. has shown that it is possible to reduce the residual tensile stress induced by grinding to a lower magnitude than the residual compressive stress that is developed by "smearing" of the surface. The result is that thin, stress-free strips are found to curve convex on the ground side, indicating residual compressive stress instead of curving concave, indicating residual tensile stress, as occurs when the strips are ground with harder wheels or with deeper cuts.

The grinding technique described by Tarasov and Grover as "gentle grinding" presumably is an approach to the General Motors Research experiments. Although these "gentle grinding" experiments are interesting and informative, it must not be assumed that "gentle grinding" can be practiced in commercial work. Commerical grinding demands methods that will not unduly hamper production. Grinding wheels of the softness that is required for "gentle grinding" reduce production rates because increased wheel wear makes it is necessary to dress the wheel more frequently. The shallow depth of cut that is necessary in "gentle grinding" cannot be tolerated in high rate production shops because of the limitations on output that would be imposed.

Since it will continue to be necessary to grind a large variety of machine parts and since "gentle grinding" as used by Tarasov and Grover is impractical in production, the best that can be expected is a compromise in the grinding practice as applied to *highly stressed parts*. The probable compromise, which is already in extensive use, will be to grind in such manner as to reduce the induced residual tensile stress to values well below the spontaneous fracture (stress-corrosion cracking) point. The ground surfaces, as well as other surfaces of the part, may then be subjected to a prestressing treatment such as shot peening, rolling or tumbling, whereby the harmful residual tensile stress is converted to beneficial residual compressive stress.

GRINDING DAMAGE REMOVED

The above procedure will result in fatigue strength of ground surfaces greater than is possible by the most "gentle" grinding alone as is indicated by the results that were obtained by Tarasov and Grover from test groups I and M, Fig. 6. In fact, the gain will probably be greater than is indicated by the above tests because a greater portion of the residual compressive stress will be retained in machine parts than in the rotating-beam fatigue specimens as will be described.

The permissible limit of residual tensile stress that may be induced by grinding can probably be measured by the etching method discussed by Tarasov in an earlier paper.[6] It must not be assumed, however, that the low-temperature annealing treatment that is recommended by Tarasov[6] is an adequate relief of the induced residual tensile stress for the purpose of restoring fatigue strength. All that this low-temperature annealing treatment can accomplish is to reduce the stress sufficiently to avoid spontaneous cracking of the ground surface before the part is put to practical use. That is, suppose that the residual tensile stress induced by grinding is 250,000 psi. and the spontaneous cracking stress after etching is 225,000 psi. A low-temperature annealing treatment may be sufficient to reduce the residual stress from 250,000 to 220,000 psi., which would prevent spontaneous cracking, but the fatigue strength of specimens that had not developed spontaneous cracks would not be improved. Adequate stress relief from the standpoint of fatigue strength is impossible in hard steel because the annealing temperature would necessarily be so great as to soften the metal. This is shown in the present paper by Tarasov and Grover which presents fatigue data in test groups I and L (Fig. 6) showing that damaging residual stress is still present after tempering for 1 hr. at 890 F.

The authors state that their observation ". . . is thought to be the first evidence that tumbling can have a beneficial effect on fatigue strength." I do not know when this effect was first observed, but it has long been well known. Tumbling has been mentioned as an alternate prestressing process in several publications.[2, 3]

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[6] L. P. Tarasov, "Detection, Causes and Prevention of Injury to Ground Surfaces," *Transactions*, Am. Soc. Metals, Vol. 36, p. 389 (1946).

ACTUAL STRESS UNKNOWN

The data presented by Tarasov and Grover purport to show the fatigue strength of specimens in terms of pounds per square inch. Since all specimens tested were residually stressed and since the magnitude of the residual stress was not measured the data should properly be presented in terms of nominal stress.

In future tests, the residual stress should be measured by processes that are capable of precision equal to or greater than is obtained by the dissection stress measurements as made by the Research Laboratories Division of the General Motors Corp. This process differs from dissection processes frequently reported in the literature only in the degree of accuracy that is considered necessary. The stress in rotating-beam, cantilever, and similar specimens is usually measured only in the direction of the principal applied tensile stress with special attention to the stress magnitude at the extreme surface. This requires the removal of many layers of metal not exceeding 0.0005 in. in thickness.[5] The special gentle grinding technique described above was developed to avoid introducing new residual stresses of appreciable magnitude, since such stresses would detract from the accuracy of the measurements.

In the absence of surface residual compressive stress of great magnitude, together with great surface yield strength such as may be developed by nitriding and carburizing, fatigue failures will always originate at the surface for the reason that the fatigue strength of surfaces is lower than sound sub-surface metal. The significant stress in fatigue is, therefore, the actual stress at the extreme surface, that is, the sum of the residual stress and the applied stress. Since fatigue failures are tensile failures, the important surface stress is that which acts in the direction of the maximum applied tensile stress.

STRESS CHANGE DURING TEST

It is not enough to measure the residual stress in specimens as processed because the magnitude of this stress changes continually during fatigue testing. The residual stress changes vary with the manner of loading and with the dynamic yield strength of the metal being tested. The dynamic yield strength is lower than the yield strength measured by static loads.

In fatigue specimens subjected to reversed bending such as were used by Tarasov and Grover, the surfaces are alternately stressed in tension and compression. Let us assume that pre-stressing as by shot peening has induced residual compressive stress equal to one half of the nominal yield strength of the metal. Let us further assume that the applied bending stress is equal to 75 per cent of the nominal yield strength of the metal. The actual strain on the compression side of the specimen will then be 50 + 75 = 125 per cent of the nominal yield strength. Compressive yielding will necessarily occur which, on the basis of the above assumptions, will reduce the residual compressive stress from 50 to 25 per cent of the nominal yield strength.

DYNAMIC YIELDING

The loss of residual stress will, however, be greater than 25 per cent because the dynamic yield strength is less than the static yield strength. Assuming that the dynamic yield strength is equal to 90 per cent of the static yield strength, we find that the retained residual compressive stress will be reduced to 15 per cent of the nominal yield strength or three-tenths of its original value. Many instances are known in which most or all of the residual compressive stress from pre-stressing operations has been lost for rotating-beam and reversed-bending specimens. In such instances the interpretation of the test data usually advanced is that the pre-stressing treatments were of little or no value. This is, of course, true for the particular test specimens but is incorrect when the treatments are applied to machine parts or to uni-directional-bending fatigue specimens.

Actual reduction of fatigue strength of shot-peened specimens is occasionally reported. These losses are found to be due to almost complete loss of the residual compressive stress by dynamic yielding. With the loss of the protective compressive stress, the stress raisers that are formed by the shot peening, such as bruises, folds, and sharp indentations, become effective in increasing local tensile stresses and early failure results.

The magnitude of the residual tensile stress that can be retained is also limited because of tensile yielding in bending, torsion, and repeated tension-fatigue fatigue tests. The residual stresses induced by severe grinding will be adjusted downward during normal fatigue testing. The fatigue damage from grinding will, therefore, automatically approach a common level providing the metal is sufficiently ductile to plastically elongate without damage.

CONVENTIONAL FATIGUE TESTS HAVE LITTLE VALUE

The loss of residual stresses from rotating-beam, reversed-bending, and reversed-torsion specimens acts to decrease the fatigue strength of specimens processed to develop residual compressive stresses. It is, therefore, important to recognize that few machine elements are loaded in such manner that the stresses are reversed, and of the few in which the load is reversed, in practically none is the stress magnitude the same in both directions.

It follows that rotating-beam, reversed-bending, and reversed-torsion specimens are incapable of measuring the worth of residual stresses in so far as these stresses will add to the fatigue strength of machine parts or to arbitrary uni-directional-loaded laboratory specimens. It is also true that our conventional tests do not measure the fatigue strength of the specimen metal; all that is measured is the fatigue weakness of the surfaces. Even this measurement is in error because the accepted procedure for preparing fatigue specimens is to cold polish the surface in the belief that this removes most of the extraneous effects and thus establishes "par" for the specimen material. It seems not to be understood that polishing induces residual compressive stress which, in the only instances in which quantitative values are available,[7, 8] increased the apparent surface strength by 11 to 15 per cent.

These facts suggest that future tests should include variously processed specimens loaded in uni-directional bending and uni-directional torsion. Interpretation of the data will require measuring the residual stress to the highest possible accuracy. The measurements should be made on specimens that have not been fatigue tested and in other specimens after prolonged fatigue testing.

MR. S. A. GORDON.[9]—I should like to ask Mr. Tarasov to describe the tumbling abrasive used. Also, was the direction of grinding the same in the flat and round bars?

The authors pointed out that the effect of shape and size was a matter of conjecture in fatigue testing. It is my

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[7] H. F. Moore, "Size Effect and Notch Sensitivity in Fatigue Tests of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, p. 516 (1945).

[8] D. Morkovin and H. F. Moore, "Effect of Size of Specimen on Fatigue Strength of Three Types of Steel," Report of the Research Committee on Fatigue of Metals (Appendix), *Proceedings*, Am. Soc. Testing Mats., Vol. 44, pp. 143, 144 (1944).

[9] Chief Structural Test Engineer, The Glenn L. Martin Co., Baltimore, Md.

opinion that substantial evidence exists to prove that size and shape have definite effects on fatigue that must be considered in the evaluation.

MR. HAAKON STYRI[10] (*by letter*).—It has been very interesting to study the tests described in this paper and the authors' evaluation of the effect of various surface finishes on flat bars in reversed bending and on round bars in rotating bending.

The values of endurance limits arrived at seem too uncertain because of the limited number of tests, particularly at loads near the endurance limit. In group D we find that more specimens have failed at longer lives than in C, yet the endurance limit for C is higher; in group F more lives are longer than in E but both have the same endurance limit. In group MJ the lives are much higher than in MG, but endurance limits are respectively 128,000 and 130,000 psi., and in group MK much higher than in MH but with only slightly higher endurance limits. It may be questioned if lives to failure at the higher loads may not be of more significance than the endurance limits in evaluating the performance of the product. That is the method used in estimating capacities of rolling bearings.

In addition to the effect of surface finish, there are other factors in the endurance of heat-treated high-carbon steels which are of importance, namely, the quality of the material and the direction of strain applied relative to grain direction.

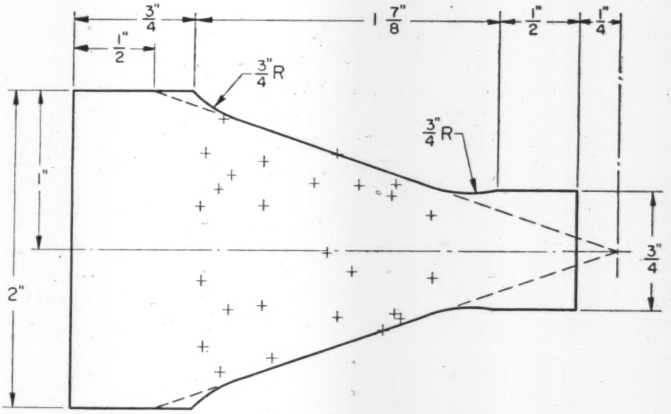

FIG. 15.—Krouse Fatigue Specimen for Sheet Metal.
+ = starting point of failure
Showing Distribution of Failures Occurring in Hardened S.A.E. 52100 Steel.

About eight years ago some tests were run in our laboratory on specimens taken from a tubing $3\frac{5}{8}$ in. in outside diameter with a $\frac{1}{2}$ in. wall, made of S.A.E. 52100 steel. From one length of tubing the outside diameter was machined down to 3.557 in. and the bore was machined to 3.432 in., so that the remaining wall would represent the outside portion of the original tube From another length of tubing the bore was machined to 2.825 in. and the outside diameter machined to 2.950 in. which gives material from the inside of the original tubing. These pieces of tubing were split lengthwise, annealed, flattened and reannealed. From the flat pieces, specimens of the Krouse type shown in Fig. 15 were blanked out, some in direction of the tube axis, others crosswise to the axis. The specimens were quenched in oil from 840 C. and tempered to 175 C. for 1 hr. They were then carefully ground on both sides to a finish of 20 microinches rms. in a direction which would be parallel to the direction of straining reversed bending, and the edges were carefully rounded by stoning. The lives are plotted in Fig. 16 and show clearly that the specimens taken from the outside of the tube and tested with the direction of the grain parallel to the direction of strain gave the best values, while the specimens from the inside of the tube and tested normal to the axis of the tube gave the lowest values.

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[10] Director of Research, SKF Industries, Inc., Philadelphia, Pa.

The conclusions drawn from this are (*a*) that harmful impurities, or other defects, are more prevalent in the bore than in the outside parts of the tube, which must be expected, as it is well known by direct observation that the core of the ingot has more impurities than the outer portion, and (*b*) that tests run with the direction of strain normal to the grain direction give lower endurance values than when the strain is applied parallel to the grain direction.

The marks + which are plotted in Fig. 15 give the location of the fractures in the specimens from all four groups, and indicate that the fractures have random starting points.

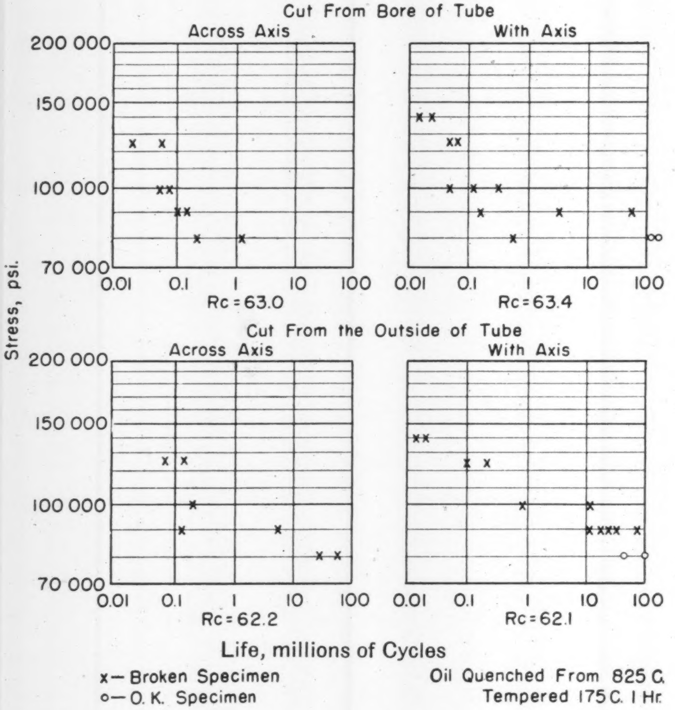

FIG. 16.—Endurance of 0.050 in. Flat Cantilever Specimens of S.A.E. 52100 Steel.

MESSRS. L. P. TARASOV AND H. J. GROVER (*authors' closure*).—Mr. Almen's interesting comments, based as they are upon considerable experience in the practical evaluation of fatigue data, include an exposition of his ideas concerning the proper way to perform fatigue tests when the effects of processes resulting in residual stresses are under investigation. However, he does not offer any experimental evidence to substantiate his conclusion that the commonly used fatigue tests, in which the specimen surfaces are stressed alternately in tension and compression, are worthless for predicting the fatigue behavior of machine parts. According to this picture, there would be little if any improvement in reversed-bending fatigue strength as a result of shot peening or tumbling, contrary to what was actually found.

His remarks deserve careful consideration in planning future tests along this line, but they do not justify his contention that we have misinterpreted many important findings because of unwarranted assumptions and failure to measure the residual surface stresses in the test specimens. The fundamental purpose of this paper was to show *what* happened to the fatigue strength of two shapes of hardened and tempered steel bars as a result of grinding or other final operation, and not *why* it happened. Granted that it would be highly interesting to relate the fatigue data to the actual residual stresses originally present in the test specimens, this is not necessary in order to find out how grinding severity, for instance, affects the fatigue strength under specified conditions of test. In this exploratory work, we chose to study the physical effects of grinding and not of grinding stresses.

We must also take exception to the statement that "extensive loss of fatigue strength is inflicted on ground machine parts," since this is bound to be misinterpreted by the casual reader. Were Mr. Almen to say "can be" instead of "is," we would be in full agreement. We do not know of any real evidence that good commercial grinding practice lowers the fatigue strength measurably, although it is true that certain processes, like shot peening, may raise it above the par value for a completely stress-free surface. Abusive grinding, which does lower it, is known from extensive trouble-shooting experience to be the exception rather than the rule. It should also be kept in mind that even when the fatigue strength is lowered by improper grinding, many parts are designed so that they do not fail from fatigue, and it is inadvisable to raise grinding costs, as might be necessary to attain perfection from the standpoint of fatigue, when the service life is considered satisfactory.

We agree that surface stresses have an important effect upon fatigue strength, as evidenced by our discussion centering about the admittedly speculative Goodman-type diagram of Fig. 11, but we do not believe that everything can be explained by stresses. The effects of cold work may be equally important. We suspect that had we measured the surface stresses as suggested, we would have found it difficult to explain some of the fatigue results in terms of the stresses alone. For example, the data in Table I show no increase in endurance limit resulting from the hand polishing of the flat bars, yet they were known to have been considerably stressed in compression by the polishing operation; at the same time, tumbled or shot-peened bars, also stressed in compression, had very much higher endurance limits.

A great deal of research will have to be performed before the effects of grinding (and other surface-affecting processes) upon the fatigue strength can be properly interpreted in terms of residual stresses, cold work, and whatever other factors may be found to be involved. The importance of obtaining detailed knowledge about grinding stresses and related phenomena is recognized by the grinding wheel manufacturers. H. R. Letner is currently studying these problems at the Mellon Institute of Industrial Research under a fellowship maintained by the Grinding Wheel Inst. When sufficient knowledge has been obtained about grinding stresses, it is considered likely that a study will then be undertaken to determine the effects of both grinding conditions and grinding stresses upon fatigue strength.

Mr. Almen's statement that "the fatigue strength of smooth specimens is reduced by an amount approaching the magnitude of the retained residual tensile stress" may be very misleading. The reduction in conventionally defined (nominal stress amplitude) endurance limits, predicted by a Goodman-type diagram such as that in Fig. 11, would be approximately:

$$S_e - S_e' = S_g S_e / S_u$$

where:

S_e = endurance limit, in psi., with no grinding stress,

S_e' = endurance limit, in psi., with grinding stress,

S_g = residual tensile stress, in psi., due to grinding, and

S_u = ultimate tensile strength, in psi.

Thus, for a steel of 300,000 psi. ultimate strength and an endurance limit of 80,000 psi., the reduction in endurance limit due to a residual tensile stress of 100,000 psi. would be about 27,000 psi. Severe grinding of the flat bars used in our investigation introduced a maximum tensile stress of about 100,000 psi., this value being estimated from stress data obtained by us in connection with other experiments. Table I shows that the endurance limit in the severely ground condition was only 15,000 to 20,000 psi. less than in the gently ground or gently ground and polished conditions.

Since Mr. Almen uses Fig. 13 to illustrate the stress gradient that may be found in a ground surface, it should be pointed out that his value of 275,000 psi. for the maximum residual tensile stress is several times higher than has been found by Frisch and Thomsen[11] for somewhat different surface grinding conditions, or by Letner,[12] and also by ourselves, for quite similar grinding conditions. Moreover, the depth of stress penetration shown in Fig. 13, even when reasonably extrapolated to zero stress, is several times lower than found by any of the investigators just mentioned. In view of the general agreement among the numerous individual determinations of grinding stress gradient, it appears that the single determination cited by Mr. Almen is not at all representative. It may be that considerable unsuspected error was introduced in his pioneering study of grinding stress gradient by the use of a honing operation to remove successive thin layers from the stressed surface in order to determine the stress distribution. The compressive stresses set up in honing may have so disturbed the changes in curvature resulting from the removal of successive layers that the calculated stresses were much too high; likewise, the compressive stresses may have masked the rather small tensile stresses present as much as several thousandths of an inch below the ground surface. It might also be remarked at this point that the ground strip in Fig. 14, used to illustrate the curvature induced by tensile surface stresses, has been ground so abusively, as shown by the outstanding burn pattern, that it should not be considered as representative of even poor commercial grinding practice.

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[11] J. Frisch and E. G. Thomsen, "Residual Grinding Stresses in Mild Steel," *Preprint 50-F-10,* Am. Soc. Mechanical Engrs. (1950).

[12] *Ibid.,* discussion by H. R. Letner.

The purpose of our "gentle grinding" was to establish a base line from which the effects of grinding severity could be conveniently determined. This gentle grinding does not measurably lower the endurance limit, and even though such grinding is not practicable under high production conditions, it is an ideal that can be approached as closely as economics warrants. We do not yet have any data to show the effect on the endurance limit of commercial grinding practice in which care is taken to avoid burning the surface, but on the basis of our unpublished flat-bar curvature data obtained under conditions representative of such practice we do not expect to find any significant drop in the endurance limit. Only further experimentation will tell just how severely a surface must be ground in order for the endurance limit to be lowered appreciably.

Mr. Almen may be right in saying saying that low-temperature stress relief of severely ground hardened steel will not restore the fatigue strength to its "uninjured" value, but we know of no evidence one way or the other. His citation of our data to support his stand is due to a misunderstanding since the tempering operation at 890 F. to which he refers was performed prior to grinding in order to lower the hardness to Rockwell C45, and not after grinding so as to stress relieve the ground surfaces.

The references cited by Mr. Almen with regard to the beneficial affect of abrasive tumbling upon fatigue strength contain no data but are unsupported assertions that tumbling is beneficial because it introduces compressive stresses. We do not consider it safe to assume that the presence of such stresses necessarily leads to an improvement in the fatigue strength. As was mentioned earlier, hand polishing of the gently ground flat bars introduced compressive stresses, conceivably of the same order of magnitude as did the tumbling operation, yet there was no increase in fatigue strength. If Mr. Almen has corroborative experimental data showing the improvement of fatigue strength as a result of tumbling, we would certainly be glad to see them published.

The stresses in the S-N diagrams were the nominal maximum applied stresses as described under "Fatigue-Test Procedures," and this should perhaps have also been stated in the diagrams themselves. However, even if we knew the magnitudes of the residual stresses, we would still use the maximum applied stresses in plotting since the residual stresses may be insufficient to define the condition of the surface with respect to fatigue strength; moreover, the residual stresses will probably be altered in the course of the fatigue test so that it would not be possible to choose a meaningful value of applied plus residual stress for plotting purposes.

While unidirectional bending may lead to different results than does reversed bending, it still does not follow that the latter method is incapable of measuring the beneficial or detrimental effects of various surface processing treatments from the standpoint of the fatigue strength of actual machine parts. Only experimentation will reveal which testing methods are reasonably satisfactory for this purpose. Unidirectional bending tests should certainly be tried, as suggested by Mr. Almen, but we have our doubts about the value of unidirectional torsion tests for evaluating the effect of grinding severity on fatigue strength for the reason that it is so difficult to obtain reproducible degrees of grinding severity in round specimens. (See discussion under *Group ML. Dry-Ground Bars*). Finally, not only should the magnitudes of the residual stresses be determined at various stages of the fatigue test but also the degree of cold working introduced by the various surface-processing treatments, if we are to understand the manner in which the treatments affect the fatigue strength.

In reply to Mr. Gordon, the tumbling abrasive consisted of electric-furnace aluminum oxide in rounded blocky form, the average dimension being in the range of $\frac{1}{2}$ to $\frac{3}{4}$ in. The grinding direction was longitudinal for both the flat and round bars. That the fatigue strength is affected by the size and shape of the test specimens is clearly indicated by the results of this and previous work, but we feel that the reason for this effect is still a matter of conjecture.

Mr. Styri brings up the point that improvement in life above the endurance limit did not correlate with improvement in endurance limit resulting from suitable changes in surface processing. A possible explanation is that not nearly enough test specimens were available to establish the sloping portions of the *S-N* curves with any degree of reliability. However, it is not necessarily surprising to find different results of surface treatment on high-stress short-lifetime behavior and on the endurance-limit behavior. Which of these regions is more important in evaluating the performance of a product depends largely on the expected service usage of this product. One purpose of presenting the complete *S-N* diagrams in the paper was to make the data available for comparison with any future results, when the factors affecting the sloping portion of the curve may be understood better than they are at present.

Since our test bars were all cut with their lengths parallel to the rolling direction, the fatigue data were not influenced by the directional effect described by Mr. Styri. Metallographic examination of the broken test bars showed only occasional inclusions, with no essential difference in cleanliness between the flat and the round bars, both of which came from the core portion of the original bar stock. Thus the inclusion content was not a factor in the pronounced difference between the fatigue data for the two types of bars.

In conclusion, the following are some of the important problems requiring detailed study if we are to understand how fatigue strength may be affected by surface processing:

1. Effect of test method, involving a comparison of unidirectional and reversed bending.
2. Effect of processing severity in terms of residual stress and cold work.
3. Effect of surface finish with respect to roughness and directional characteristics.

EFFECT OF CHROMIUM PLATING ON THE ENDURANCE LIMIT OF STEELS USED IN AIRCRAFT*, [1]

By Hugh L. Logan[2]

Synopsis

Chromium plating reduced the endurance limits of both normalized and hardened (quenched-and-tempered) S.A.E. X4130 steels; the reduction was larger for the hardened steel. The endurance limits for steel of a given hardness decreased with increased plating bath temperatures. Baking of the plated steel, at temperatures up to 350 C., reduced the endurance limit; baking at 440 C. increased the endurance limit of the plated steel. However (for hardened steel) baking did not restore the endurance limit to that of the unplated steel. Damaging effects of chromium on the endurance limits of plated steels are attributed to stresses in the chromium or steel, which are increased by low-temperature baking but are relieved in part by baking at 400 to 440 C.

During the period of 1930 to 1936 the Bureau of Aeronautics, Navy Department, submitted for examination a number of chromium-plated hollow-steel airplane propeller blades that had failed by fatigue in service. Preliminary fatigue tests in the laboratory confirmed conclusions, based on the examination of the propeller failures, that the endurance limit of the steel was materially reduced by chromium plating. In addition to the plating of propeller blades and other parts for protective purposes, manufacturers and maintenance shops were exploring the possibilities of reclaiming by chromium plating worn and undersize parts. These considerations prompted the Bureau of Aeronautics to support an extensive investigation conducted by the National Bureau of Standards on the effect of chromium plating on the endurance limits of steels used in aircraft.

Two steels designated S.A.E. X4130 and 6130 were used in the investigation. However, most of the tests were made on X4130 steel, heat treated to a hardness of about Rockwell 40 C, with an ultimate tensile strength of approximately 180,000 psi.

The first phase of this study provided for the determination of the effects of chromium, as plated, on the endurance limit of the steels. The variables studied were: (1) plate thickness, (2) hardness of the steels plated, (3) current densities and temperatures of the plating baths, (4) surface grinding of the chromium after plating, and (5) interruptions of the plating process.

A second phase of the investigation was a systematic study of the effect of heating on the endurance limit of specimens of chromium-plated X4130 steel. If the decrease in the endurance limit of the plated steel is attributable to the large quantities of hydrogen evolved at the cathode during the process of chromium plating, then it appears that the endurance limit may be increased by the removal of hydrogen by heating. However, Swanger and France (**1**)[3] found that heating the specimens for 5 hr. at 350 F. (182 C.) reduced the endurance limit of the plated steel to about 70 per cent of that of the plated but unheated material. Wiegand and Scheinost (**2**) also reported that heating of chromium-plated steel at 250 C. decreased its endurance limit. Other workers have spot checked the effect on the endurance limit of heating chromium-plated steel. However, no systematic study of the effect of heating had been made.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[1] Reprinted from *Journal of Research*, Nat. Bureau Standards, Vol. 43, August, 1949.

[2] Metallurigist, National Bureau of Standards, Washington, D. C.

TABLE I.—COMPOSITION OF STEELS.

Element	S.A.E. X4130 Steel			S.A.E. 6130 Steel
	Lot A	Lot B	Lot C	
Carbon, per cent	0.35	0.29	0.33	0.30
Manganese, per cent	0.53	0.47	0.55	0.61
Phosphorus, per cent	0.01	0.02	0.03	0.03
Sulfur, per cent	0.02	0.02	0.01	0.04
Silicon, per cent	0.21	0.25	0.24	0.25
Chromium, per cent	0.95	0.96	1.05	1.01
Molybdenum, per cent	0.19	0.24	0.20	[a]
Vanadium, per cent	[a]	[a]	[a]	0.22
Nickel, per cent	[a]	0.15	0.17	[a]

[a] Not determined.

A theory has been developed to explain, at least in part, the effects of chromium plating on the endurance limit of the steels studied.

Publication of this material, including data circulated during wartime in reports (**3**) that at that time were restricted but have since been declassified, has been approved by the Bureau of Aeronautics, sponsor of the investigation.

MATERIALS

The materials, from which the specimens for this investigation were machined, consisted of three lots of S.A.E. X4130 rod and one lot of 6130 rod. The compositions of these steels, as determined by chemical and spectrochemical analyses, are shown in Table I.

Lot A of the X4130 steel was supplied by the Naval Aircraft Factory in two heat-treated conditions, normalized to Rockwell hardness 89 B, and quenched and tempered to Rockwell 39 C. The 6130 steel was also supplied by the Naval Aircraft Factory but only in the quenched-and-tempered condition, Rockwell 33 C. The other lots of X4130 rod were heat treated at the National Bureau of Standards; they were quenched in oil from approximately 1600 F. and subsequently tempered to about Rockwell 40 C.

Typical microstructures of transverse sections of the steels are shown in Fig. 1. A longitudinal section of the normalized X4130 steel (not illustrated) showed definite indications of banding and segregation, which are not unusual for this type of steel.

SPECIMENS AND TEST METHODS

Preparation and Testing of Specimens:

Specimens for the fatigue tests were of the usual R. R. Moore type (**4**) with nominal diameters of 0.30 in. in most cases; however, 0.25-in.-diameter specimens were used in some instances. Computation of the stresses in the outer fibers of the specimens, and hence the endurance limits, were based on the diameters of the specimens prior to plating. Diameters of all specimens machined at this Bureau were determined, before and after plating, with a dial gage comparator reading to 0.0001 in.

Prior to the final polishing, the specimens prepared at this Bureau were ground with a Norton "38" alundum wheel. Subsequently these grinding marks were removed with metallographic polishing papers by one of the following methods: (*a*) circumferential polishing of specimens in a lathe for fixed periods, determined by experiment to be sufficient to remove grinding marks or marks of coarser papers previously used (papers used were Behr-Manning emery 1, Aloxite 400, Behr-Manning emery 0 and 00); or (*b*) longitudinal polishing of specimens using Aloxite 400 paper followed by the Behr-Manning emery 0 polishing paper in the form of belts mounted on a rubber-backed wheel. In some instances, commercially available coarse and medium emery-impregnated rubber wheels were used in place of the abrasive paper belts.

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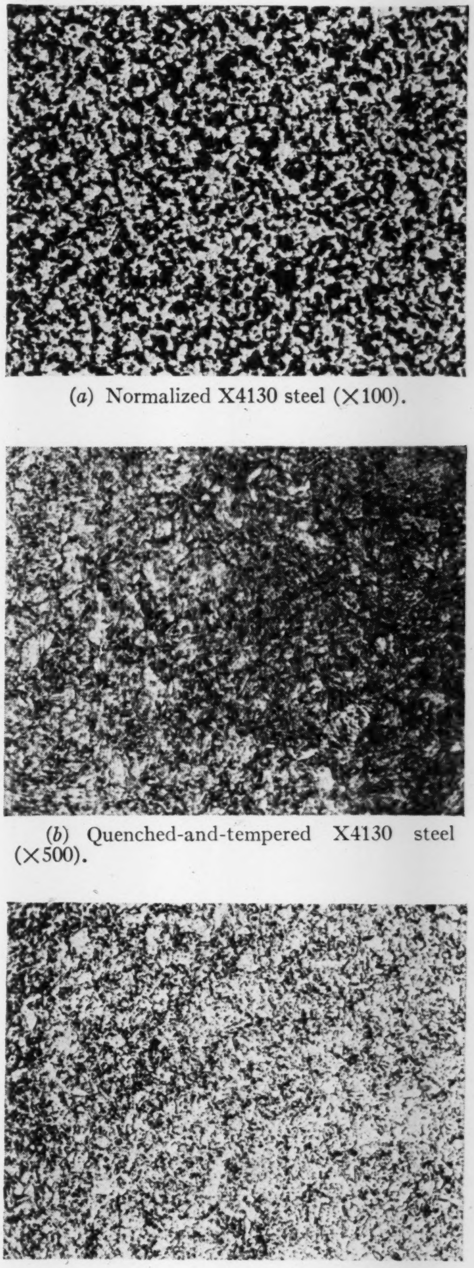
[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 712.

A few of the mechanically polished specimens were subsequently polished electrolytically as follows: Specimens were made the anodes in a bath containing 50 per cent by volume of concentrated H_2SO_4 and 50 per cent by volume of 75 per cent H_3PO_4; a current density of 250 amp. per sq. ft. was maintained for 5 min.; the temperature was 42 C. This reduced the diameters of the specimens by 0.0004 to 0.0006 in. Details regarding the preparation of the fatigue specimens are given in Table II.

Fatigue tests were made in R. R. Moore type rotating-beam fatigue testing machines operating at 1800 or 3600 rpm. with stresses generally applied in increments of 1000 psi. Generally, eight to ten specimens were required to obtain the endurance limit in any one case. Specimens that had run 10,000,000 or more cycles without failure were considered to have been stressed at or below the endurance limit and were removed from the machines.



(*a*) Normalized X4130 steel (×100).

(*b*) Quenched-and-tempered X4130 steel (×500).

(*c*) Quenched-and-tempered 6130 steel (×500).

FIG. 1.—Typical Microstructures of Transverse Sections of Steels Investigated. All Etched in 1 per cent Nital.

Electroplating of Specimens:

Specimens were plated at the National Bureau of Standards by personnel of the Electrodeposition Section. The process prior to plating was as follows: Specimens were cleaned by scrubbing with fine pumice powder and rinsed thoroughly with water. The light oxide film that formed during the time required to assemble the specimens in the plating rack was removed by dipping for a few seconds in 20 per cent hydrochloric acid solution. The specimens were again rinsed, placed in the chromium plating solution, and made anodic for 1 min. at the same current density used in plating. After the anodic treatment, full plating current was immediately applied.

TABLE II.—DETAILS OF PREPARATION OF SPECIMENS USED IN THE INVESTIGATION.

S.A.E. No.	Steel Lot	Heat Treatment	Rockwell Hardness	Specimens Prepared for Plating by[a]	Specimens Chromium Plated by	Temperature of Plating Bath, deg. Cent.	Plating Current Density, amp. per sq. ft.	Nominal Thickness of Plating, in.
X4130	A	....	89 B	NAF	NAF	55	200	0.0001; 0.001; 0.004; 0.009[b]
X4130	A	....	89 B			70	1000	0.0001; 0.001; 0.004; 0.009[b]
X4130	A	....	39 C			55	200	0.0001; 0.001; 0.004; 0.009[b]
X4130	A	....	39 C			70	1000	0.0001; 0.001; 0.004; 0.009[b]
6130	....	....	33 C			55	200	0.0001; 0.001; 0.004; 0.009[b]
6130	....	....	33 C			70	1000	0.0001; 0.001; 0.004; 0.009[b]
X4130	B	Oil quenched from 1575 to 1600 F. drawn at 900 to 925 F.	40 C	NBS	NBS	70	1000	0.009
X4130	C		40 C			70	1000	0.009
X4130	C		40 C			55	350	0.001; 0.005; 0.010; 0.017
X4130	C		40 C			85	700	0.010

[a] Includes heat treating, machining, and usually polishing.

[b] Two groups of specimens had been plated to a thickness of about 0.008 in. One of these groups was stated to have been plated to a thickness of 0.010 in. with about 0.001 in. of plate subsequently removed by grinding.

The plating electrolyte contained 250 g. per liter of CrO_3 and 2.5 g. per liter of H_2SO_4. Specimens were plated at three different bath temperatures and current densities. A bath temperature of 55 C. and a current density of 350 amp. per sq. ft. were selected as one condition, because they were within the range (45 to 60 C. and 70 to 450 amp. per sq. ft.) generally used in industrial chromium plating. A temperature of 70 C. and a current density of 1000 amp. per sq. ft. were employed for the second condition in order to check certain results previously obtained in this investigation on specimens similarly plated at the Naval Aircraft Factory. A bath-temperature of 85 C. and a current density of 700 amp. per sq. ft. were selected for the third condition, since chromium deposited in this way is supposed to be subject to less contraction when heated than is chromium plated under the other conditions, and it appeared advisable to determine the extent to which this factor might affect the endurance limit of the steel.

In order to determine the effect of interrupted plating on the endurance limit, two sets of specimens were plated to the same final thickness; one set being plated continously and the other by an interrupted operation. Plating was resumed following the interruptions only after the employment of the proper commercial technique for plating chromium on chromium; this involved a short reversal of the current and the gradual increasing of the current from a low value to the plating current.

No information was provided by the Naval Aircraft Factory as to the surface preparation, after the final machining and prior to plating, of fatigue specimens prepared at that agency. The composition of the plating bath used by the Naval Aircraft Factory in plating of fatigue specimens was the same as that used at this Bureau and described above; specimens were stated to have been plated in a bath maintained at 55 C. at a current density of 200 amp. per sq. ft. and at 70 C. and 1000 amp. per sq. ft.

TABLE III.—ENDURANCE LIMITS OF UNPLATED AND CHROMIUM-PLATED STEELS.

Steel[a]	Nominal Plating Thickness, in. 0 in.	0.0001 in.		0.001 in.		0.004 in.		0.009 in.		Ground to 0.009 in.		0.017 in.	
	Endurance Limit, psi.	Endurance Limit, psi.	Percentage of Unplated Steel	Endurance Limit, psi.	Percentage of Unplated Steel	Endurance Limit, psi.	Percentage of Unplated Steel	Endurance Limit, psi.	Percentage of Unplated Steel	Endurance Limit, psi.	Percentage of Unplated Steel	Endurance Limit, psi.	Percentage of Unplated Steel
PLATED AT 70 C.—CURRENT DENSITY 1000 AMP. PER SQ. FT.													
Normalized X4130. Hardness, Rockwell 89 B	44 000	36 500	83	33 000	75	37 000	84	40 000	91	38 000	86	....	....
Q & T 6130. Hardness, Rockwell 33 C	83 000	48 000	58	47 000	57	51 000	61	55 000	66	55 000	66	....	....
Q & T X4130. Hardness, Rockwell 39 C	93 000	59 000	63	61 000	66	50 000	54	45 000	48	58 000	62	....	....
PLATED AT 55 C.—CURRENT DENSITY 200 AMP. PER SQ. FT.													
Normalized X4130. Hardness, Rockwell 89 B	44 000	37 000	84	41 000	93	42 000	95.5	40 000	91	45 000	102	....	....
Q & T 6130. Hardness, Rockwell 33 C	83 000	64 000	77	65 000	78	60 000	72	56 000	67	61 000	74	....	....
Q & T X4130. Hardness, Rockwell 39 C to 40 C	93 000	73 000	79	69 000	74	67 000	62	49 000	53	65 000	70	....	....
PLATED AT 55 C.—CURRENT DENSITY 350 AMP. PER SQ. FT.													
Q & T X4130. Hardness, Rockwell 39 C to 40 C	93 000	....	....	66 000	71	....	....	64 000	69	....	....	61 000	66
PLATED AT 85 C.—CURRENT DENSITY 700 AMP. PER SQ. FT.													
Q & T X4130. Hardness, Rockwell 39 C to 40 C	93 000	....	....	....	....	....	....	10 000	11	....	....	....	....

[a] Q & T = Quenched and tempered.

Heat Treatment of Chromium-Plated Specimens:

Some specimens were heated in oil at about 100 C., in boiling ethylene glycol at 193 C., or in N-butal phthalate at 297 C. Other specimens were heated to temperatures up to 200 C. in an electrically-heated laboratory oven, with forced air circulation, thermostatically controlled to ±2 C. Temperatures of 300, 350, 400, and 440 C., controlled to ±3 C., were obtained with an electrically-heated tempering furnace with forced air circulation.

RESULTS AND DISCUSSION

Effect of Chromium Plating on the Endurance Limits of S.A.E. X4130 and S.A.E. 6130 Steels:

Normalized S.A.E. X4130 Steel.—The results of fatigue tests on the normalized X4130 steel (heat A) are given in Table III and shown graphically in Fig. 2. The decrease in the endurance limit of the normalized steel caused by chromium plating was generally small; the minimum endurance limit of any group of plated specimens was 75 per cent of the endurance limit of the unplated steel. This value was obtained on specimens plated to a thickness of 0.001 in. at a current density of 1000 amp. per sq. ft. Most sets of specimens had endurance limits ranging from 83 to 95.5 per cent of that of the unplated steel. Specimens plated in the bath at 55 C. and a current density of 200 amp. per sq. ft. had endurance limits equal to or greater than those plated at 70 C. and 1000 amp. per sq. ft.

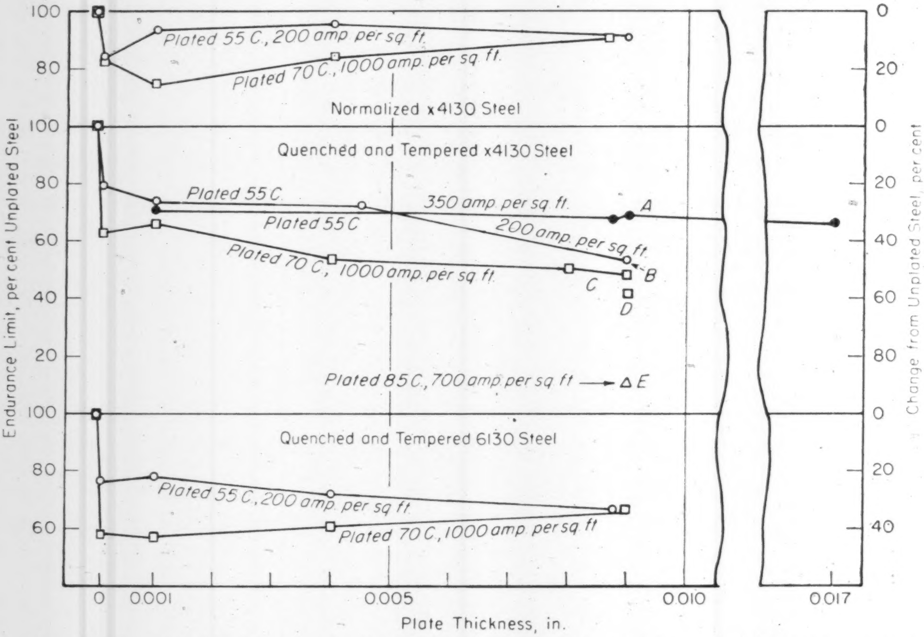


FIG. 2.—Relationship Between the Endurance Limits and Plate Thicknesses of X4130 and 6130 Steels.

Data obtained on specimens plated at National Bureau of Standards are shown by curve *A* and points *D* and *E*. All other data were obtained on specimens plated at the Naval Aircraft Factory

Quenched and Tempered S.A.E. X4130 Steel.—The results of fatigue tests on quenched-and-tempered S.A.E. X4130 steel (lots A, B, and C), hardness approximately 40 Rockwell C, bare, and plated to various thicknesses under various plating conditions are given in Table III. Most of the data are also shown graphically in Fig. 2. The endurance limits of the quenched-and-tempered steel specimens were reduced much more by chromium plating than were those of the normalized specimens (hardness, Rockwell 89 B). The maximum endurance limits of specimens plated to a thickness of 0.008 to 0.010 in. at a bath temperature of 55 C. and tested as plated were 68 to 69 per cent of that of the unplated steel. Endurance limits of the specimens in general decreased with increased bath temperatures. Specimens plated at 70 C. to the same thickness as those plated at 55 C. and discussed above had endurance limits 40 to 48 per cent of that of the unplated steel, and those plated at 85 C. only 11 per cent of that of the unplated steel.

Endurance limits of specimens of the quenched-and-tempered steel, plated at the Naval Aircraft Factory (Table II), generally decreased with increased plate thickness (Fig. 2, curves *B* and *C*). There was no significant change in endurance limit with increased plate thickness for specimens plated at 55 C. at the National Bureau of Standards (Fig. 2, curve *A*). Plating at this Bureau in the bath at 55 C. was at a current density of 350 amp. per sq. ft. as compared to a current density of 200 amp. per sq. ft. at that temperature at the Naval Aircraft Factory. Specimens plated by the Naval Aircraft Factory to a thickness of approximately 0.009 in. at 70 C., current density 1000 amp. per sq. ft., had a 14 per cent higher endurance limit than specimens plated under the same conditions at this Bureau. Curve *C*, Fig. 2, represents Naval Aircraft Factory specimens, and point *D*, National Bureau of Standards specimens.

TABLE IV.—ENDURANCE PROPERTIES OF S.A.E. X4130 STEEL SPECIMENS (HARDNESS, APPROXIMATELY ROCKWELL 40 C) CHROMIUM PLATED AND SUBSEQUENTLY HEATED.

Nominal Thickness of Plating, in.	Heat Treatment			Endurance Limit	
	Temperature, deg. Cent.	Medium	Time, hr.	psi.	Percentage of Unplated Steel
LOT C STEEL, PLATING BATH TEMPERATURE 55 C., CURRENT DENSITY 350 AMP. PER SQ. FT.					
0.0085	....	None	....	64 000	67
0.009	100	Oil	1	56 000	64
0.010	100		12	<48 000	<55
0.0095	193	Ethylene glycol	1	35 000	40
0.0105	193		6	33 000	37.5
0.0100	193		100	33 000	37.5
0.0105	193	Air	1	24 000	27
0.0085	200		6	32 000	34
0.0087	296	Butal phthalate	1	35 000	40
0.009	440	Air	1	83 000	87.5
LOT B STEEL, PLATING BATH TEMPERATURE 70 C., CURRENT DENSITY 1000 AMP. PER SQ. FT.					
0.008	....	None	....	45 000	51
0.008	95.5	Oil	24	39 000	44
0.008	95.5	Air	24	35 000	40
0.008	200		6	32 000	36
0.008	300		4	46 000	52
0.008	350		1	51 000	58
0.008	400		1	55 000	63
0.008	440		1	59 000	67
LOT C STEEL, PLATING BATH TEMPERATURE 70 C., CURRENT DENSITY 1000 AMP. PER SQ. FT.					
0.009	....	None	....	40 000	42
0.008	200	Air	6	25 000	26
0.008	300		4	28 000	28.5
0.008	440		1	47 000	49.5
LOT C STEEL, PLATING BATH TEMPERATURE 85 C., CURRENT DENSITY 700 AMP. PER SQ. FT.					
0.010	....	None	....	10 000	11
0.010	193	Ethylene glycol	6	13 000	14.5
0.010	440	Air	1	42 000	46.5

There was no significant difference in the endurance limits of two sets of specimens, both plated to the same final thickness under similar conditions, of which one was plated continuously and the other by an interrupted operation.

Quenched-and-Tempered S.A.E. 6130 Steel.—The results of fatigue tests on the quenched-and-tempered S.A.E. 6130 steel (Rockwell hardness 33 C), bare and plated to various thicknesses at current densities of 1000 and 200 amp. per sq. ft., are given in Table III and are shown graphically in Fig. 2.

Endurance limits of specimens plated at 70 C. increased as the plating thickness was increased above 0.001 in.; on the other hand, the endurance limits of specimens plated at 55 C. decreased with increased plate thickness above that value. The endurance limits of specimens with 0.009-in. thick plating were approximately the same for both sets of plating conditions and amounted to 66 to 67 per cent of that of the unplated steel. Specimens plated to thicknesses of 0.0001 and 0.001 in. had endurance limits of 77 to 78 and 57 to 58 per cent of that of the unplated steel for bath temperatures of 55 and 70 C., respectively.

The data obtained on the ground and unground specimens having approximately the same final plate thickness (0.009 in.), in general, indicated that the ground specimens had endurance limits equal to or greater than those of the "as-plated" specimens (Table III).

Effect of Heating on the Endurance Limit of Chromium-Plated S.A.E. X4130 Steel:

The results of fatigue tests on specimens of lots B and C of the S.A.E. X4130 steel, chromium plated to thicknesses of 0.001 to 0.017 in. and subsequently baked at temperatures ranging from 100 to 440 C., are given in Tables IV, V, and VI; some of the data are shown graphically in Figs. 3 and 4. The endurance limits of these specimens reached a minimum value for temperatures between 193 and 300 C.; at higher baking temperatures the endurance limits increased, and at temperatures of 400 and 440 C. exceeded those of the unbaked plated specimens. The endurance limit of one set of specimens heated in air for 1 hr. at 193 C. was 27 per cent of that of the unplated steel (Table IV). The endurance limit of specimens heated in ethylene glycol at 193 C. was somewhat higher than that of specimens heated in air and was independent of the time specimens were held at the baking temperature. Specimens plated to a thickness of 0.002 in. and heated for 1 hr. at 440 C. had an endurance limit as high as 87½ per cent of that of the unplated steel compared to 68 to 69 per cent for unbaked plated specimens. However, the endurance limits of the plated specimens heated 1 hr. at 440 C. decreased as the ratio of the cross-sectional area of chromium plating to steel increased, reaching values less than for the unbaked steel for very thick plating.

TABLE V.—EFFECT OF SPECIMEN DIAMETER AND PLATING THICKNESS ON THE ENDURANCE LIMIT OF QUENCHED-AND-TEMPERATED S.A.E. X1430 STEEL, HARDNESS ROCKWELL, 40 C. LOT C, CHROMIUM PLATED AT A CURRENT DENSITY OF 350 AMP. PER SQ. FT., BATH TEMPERATURE 55 C.

Specimen Diameter, in.	Nominal Plate Thickness, in.	Ratio of Cross-Sectional Chromium Plate to Steel Area	Baking Treatment		Endurance Limit, psi.	Percentage of Endurance Limit of Unplated Steel
			Time, hr.	Temperature, deg. Cent.		
0.25 to 0.30	....	0	....	....	95 000	100
0.30	0.017	0.24	....	....	63 000	66
0.30	0.0085	0.116	....	....	64 000	67
0.30	0.001	0.014	....	....	66 000	69
0.25	0.0105	0.22	....	....	57 000	60
0.25	0.005	0.090	....	....	59 000	62
0.25	0.001	0.021	....	....	59 000	62
0.30	0.0085	0.116	2	200	31 000	33
0.30	0.0085	0.116	6	200	31 000	33
0.30	0.018	0.265	1	440	<55 000	<58
0.25	0.002	0.122	1	440	83 000	87.5
0.25	0.011	0.184	1	440	61 000	64
0.25	0.010	0.164	1	440	67 000	71
0.25	0.009	0.157	2	440	80 000	84
0.25	0.012	0.201	4	440	70 000	74
0.25	0.005	0.082	1	440	80 000	84
0.25	0.0015	0.024	1	440	82 000	86

TABLE VI.—EFFECT OF TYPE OF SURFACE PREPARATION OF THE BASIS STEEL PRIOR TO PLATING, ON THE ENDURANCE PROPERTIES OF S.A.E. X4130 STEEL (LOT C) CHROMIUM PLATED TO A THICKNESS OF 0.008 TO 0.009 IN. AT A CURRENT DENSITY OF 1000 AMP. PER SQ. FT.

Bath temperature 70 C.

Type of Surface Treatment[a]	Heat Treatment		Endurance Limit, psi.	Percentage of Endurance Limit of Unplated Mechanically Polished Steel
	Time, hr.	Temperature, deg. Cent.		
Mechanical[b]	....	....	95 000	100
Electrochemical[b]	....	....	87 000	92
Mechanical	....	....	40 000	42
Electrochemical	....	....	24 000	25
Mechanical	6	200	25 000	26.5
Electrochemical	6	200	24 000	25
Mechanical	1	440	47 000	49.5
Electrochemical	1	440	45 000	47.5

[a] All specimens were mechanically polished in a longitudinal direction with "400A" Aloxite paper. Subsequently, specimens labeled "Electrochemical" were made anodes in $H_2SO_4 + H_3PO_4$ solution and were reduced about 0.0005 in. in diameter by electrochemical means.

[b] Not plated.

The endurance limit of specimens polished electrolytically prior to plating and tested without subsequent heating was lower than that of mechanically polished specimens (Table VI). However, the endurance limits of the plated and heated specimens were independent of the original surface preparation prior to plating.

Discussion of Causes of Effects of Chromium Plating on Endurance Limits of Steels:

Three possible explanations for the fact that the endurance limits of the steels were reduced by the chromium plate have been proposed: The embrittlement due to hydrogen; cracks in the chromium plate at which stress concentrations occur; and the presence of residual stresses in the chromium or steel or both as the result of the plating. It is of course recognized that these factors may be interrelated and possibly be manifestations of one another.

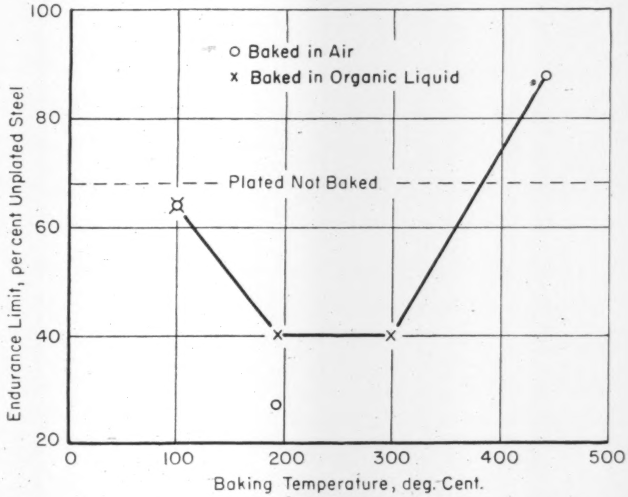


FIG. 3.—Influence of Baking Temperatures on the Endurance Limits of Quenched-and-Tempered X4130 Steel Specimens Chromium Plated at 55 C. and a Current Density of 350 amp. per sq. ft. to a Thickness Approximately 0.008 in.

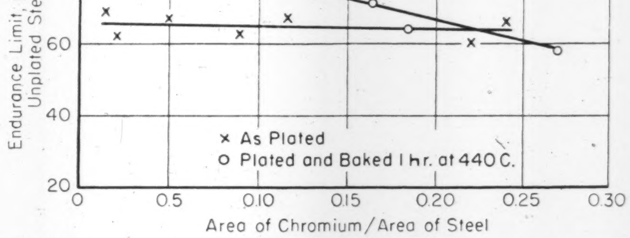


FIG. 4.—Relationship Between Endurance Limit and Ratio of Cross-Sectional Chromium-Plated Area to Steel Area for Quenched-and-Tempered S.A.E. X4130 Steel Specimens 0.250 and 0.300 in. in Diameter.

It is well known that the plating efficiency for depositing chromium is low, approximately 15 to 20 per cent of the current being used to deposit chromium. Large quantities of hydrogen are produced at the cathode, and the chromium plate has been shown to contain as much as 128 volumes of hydrogen per volume of chromium (5, 6). Theories explaining the manner in which the cathodic hydrogen is held in the chromium (7) have been proposed but are not pertinent to the discussion at this point. Zapffe (8) has explained brittleness observed in bend tests on wire plated with chromium as due to hydrogen. Brenner, Burkhead, and Jennings (9) showed that approximately 95 per cent of the hydrogen is removed by heating chromium at 450 C. These facts suggest that the improvement in endurance limit of specimens heated at 440 C. is due to the expulsion of hydrogen. However, specimens heated at 200 C. had in some cases only 50 per cent of the endurance limit of unheated specimens, whereas data given by Brenner and co-workerss howed that approximately half of the hydrogen was removed from the chromium by heating at 200 C. It is difficult, therefore, to see how the results of the fatigue tests reported here can be attributed directly to hydrogen embrittlement.

At a magnification of 250 diameters, the surfaces of the electroplated chromium samples studied had a pebble-grained appearance. The specimens plated at 55 and 70 C. also exhibited surface cracks having the appearance of grain boundaries (Figs. 5 and 6). The cracks were less numerous in the specimens plated at 70 C. than for those plated at the lower temperature. No cracks were found on specimens plated at 85 C. Metallographic examinations of longitudinal sections of the chromium-plated specimens at distances of 0.01 in. or more from the fatigue fracture failed to reveal cracks penetrating through the chromium, or to reveal cracks extending any appreciable distance into the chromium from the outer surface. There were, however, numerous inclusions and voids in the chromium.

Mehr, Oberg, and Teres (10) reported that, in general, the number of cracks in a given area increased as the chromium plate was heated. Other observers (3) had noted that heating of the chromium plate at a temperature as low as 100 C. tended to widen the cracks. The author found this crack-widening tendency to be more pronounced as the temperature of heating was increased (Fig. 7).

Bennett (11) has shown that notches reduce the endurance limit of normalized X4130 steel approximately 20 per cent and that fine cracks reduce the endurance limit by more than 50 per cent. The presence of cracks in the chromium plated at 55 and 70 C. might explain the reduction in the endurance limits of steel specimens plated at these temperatures. However, no cracks were found in chromium plated at 85 C., and specimens plated at this temperature had the lowest endurance limit of any specimens tested. Furthermore, preliminary results of an investigation, now in progress, indicate that fatigue damage in chromium-plated steel specimens starts below the surface of the chromium and does not appear to be associated with the surface cracks developed during the plating of the chromium. It must be concluded, therefore, that cracks in the chromium do not have the same effect on the endurance limit of specimens as cracks in the basis steel.

It is well established that residual stresses in a metal markedly influence its endurance limit. The increased endurance limits, obtained by the shot peening (12) of steel parts, are believed to be the result of residual compressive stresses in the surface of the metal. Conversely, residual tensile stresses in the surface layers of the specimens are considered to lower the endurance limit of the material. Hume-Rothery (13) reported tensile stresses as high as 50,000 psi. in electrolytically deposited chromium. Brenner, Burkhead, and Jennings (9) reported that the residual tensile stresses in chromium plated to a thick-

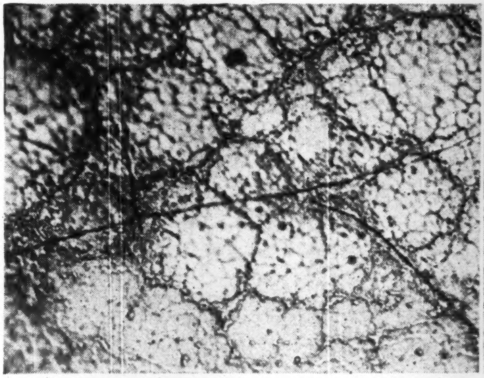


FIG. 5.—Surface of Chromium Plated on Steel at 70 C. (Current Density 1000 amp. per sq. ft.). Unetched, ×500.

Areas bounded by sharply defined cracks are larger than those in chromium plated at 55 C. (see Fig. 6).

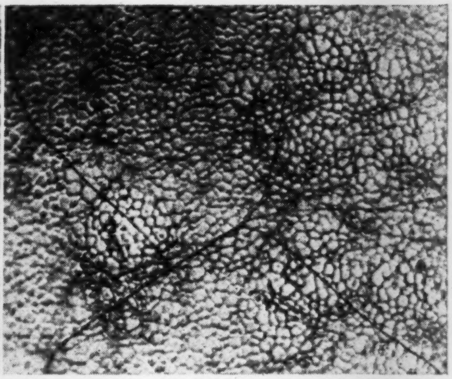
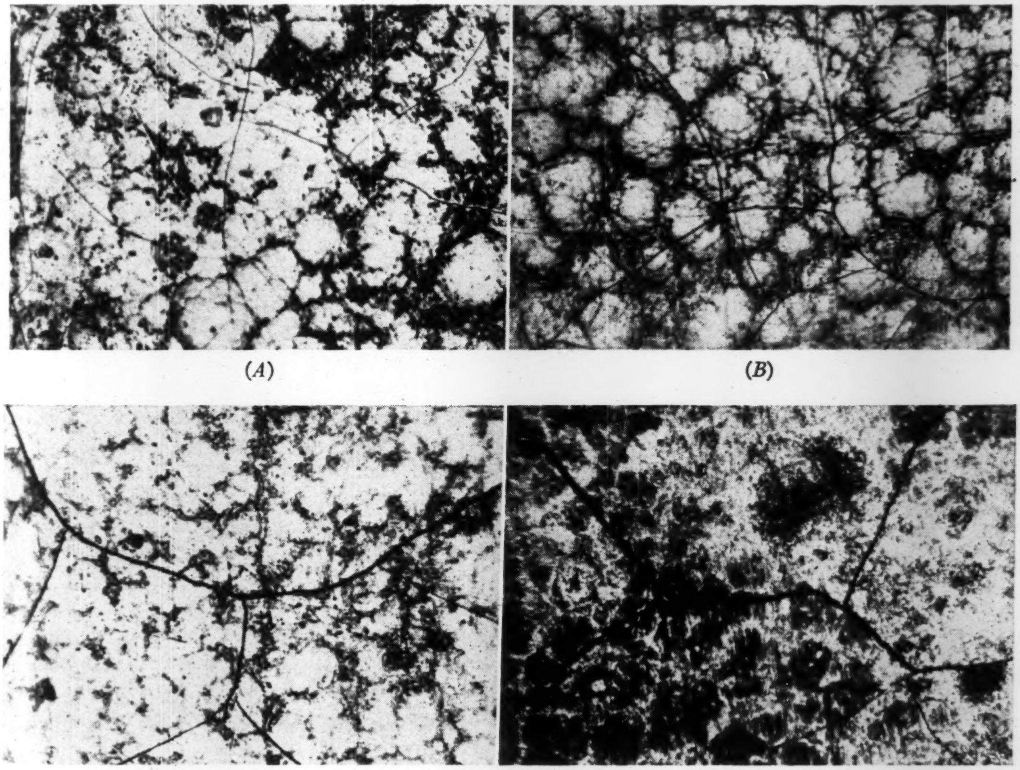


FIG. 6.—Surface of Chromium Plated on Steel at 55 C. (Current Density 200 amp. per sq. ft.). Unetched, ×500.



(*A*) (*B*)

(*C*) (*D*)

FIG. 7.—Cracks in Chromium Plated at 55 C. (350 amp. per sq. ft.). Unetched, ×250.

A, as plated; *B*, heated 1 hr. at 193 C.; *C*, heated 1 hr. at 296 C.; *D*, heated 1 hr. at 440 C.

ness of 8×10^{-4} in. increased from approximately 15,000 psi. for a bath temperature of 50 C. to over 60,000 psi. for chromium plated from a bath at 85 C. The endurance limit of specimens plated at 55 C. was higher than that of specimens plated at 70 C. and very much higher than that of specimens plated at 85 C. This is to be expected if the internal stress in the chromium plate increased with the temperature of the plating bath in accordance with Brenner's findings.

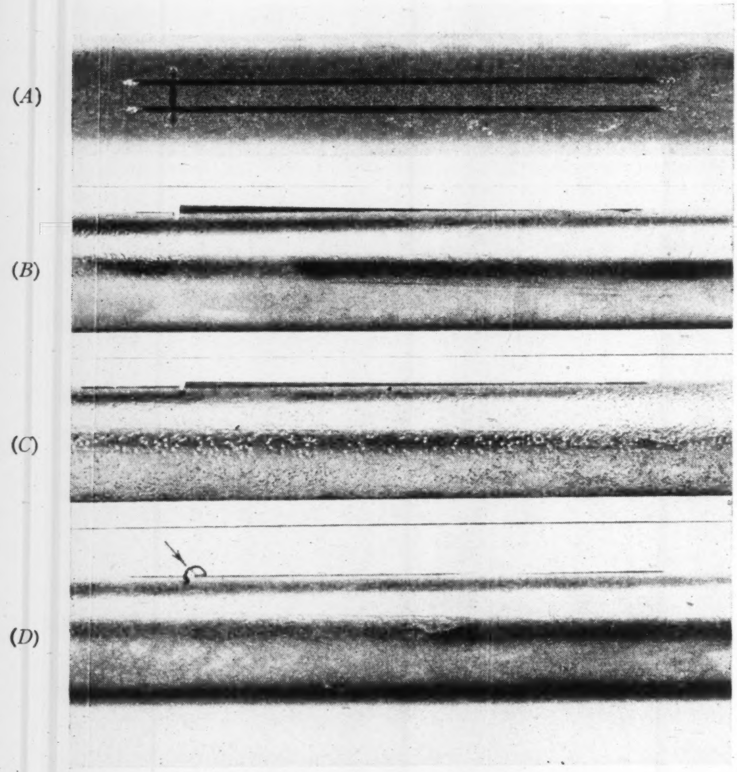


FIG. 8.—Chromium-Plated Steel Tubes.

A, Top view of tube showing "tongue," *B*, *C*, and *D*, side views of tubes showing deflections of "tongue." *B*, tube plated, heated at 200 C. and "tongue" subsequently cut; *C*, tube plated, "tongue" cut and tube subsequently heated at 200 C.; *D*, tube plated, heated at 440 C. and "tongue" cut. A $\frac{3}{16}$-in. ball bearing (indicated by arrow) is resting on the depressed "tongue" surface.

The formation of cracks in chromium plating is usually attributed to the rupture of the deposit as the result of high residual tensile stresses (9). The presence of cracks in the chromium, therefore, indicated at least partial relief of residual stresses. Specimens having the largest number of cracks per unit area, discussed above, would be expected to have the lowest residual stresses, and the residual stresses would be greater as the cracks per unit area decreased (and as the plating temperature increased).

Hidnert (14) showed that an electrodeposited chromium specimen, on the first heating to 500 C., decreased in length and that during the cooling to room temperature it continued to decrease in length. The net decrease in length amounted to 1.1 per cent. Hidnert's results showed an inflection in the heating curve at about 300 C. Preliminary results of a current investigation by the author indicate that there is an inflection in the temperature - electrical resistivity curve at about this same temperature. Hidnert did not give any data for specimens heated to 200 C. and cooled to room temperature. In the present investigation, the densities of tubes of chromium were determined for each of the following conditions: "as deposited," after heating at 193 C. (in boiling ethylene glycol) and after a second heating in helium at 440 C. The densities were 6.95 g. per cu. cm. as plated, 7.01 g. per cu. cm. after heating at 193 C., and 7.09 g. per cu. cm. after heating at 440 C. Brenner (9) indicated that changes in the volume of chromium on heating were isotropic. Hence, heating at 200 C. would tend to reduce the volume of the plated chromium. If the chromium was restrained from contracting, as for example by being plated on steel, residual tensile stresses would be set up in the chromium that might be expected to lower the endurance limit. That such stresses do exist in chromium plated on steel is shown in Fig. 8 (*B* and *C*). Thin-walled steel tubes were annealed, pickled to remove scale, and chromium plated. Tube *B*, Fig. 8, was heated at 200 C. for 6 hr., and a "tongue" was subsequently cut in the tube. The deflection shown indicated that there had been residual tensile stresses in the chromium. The tongue was cut in tube *C* after plating and was deflected only a small amount. Heating at 200 C., however, caused the chromium to contract in volume and produced the deflection shown.

Heating of chromium at a temperature of 440 C. produced, as indicated by the density data, more than twice the contraction obtained by heating at 193 C. It is suggested that if a chromium-plated steel specimen is heated to 440 C., the concurrent expansion of the steel and contraction of the chromium stresses the chromium above its ultimate tensile strength at that temperature, consequently relieving the internal stresses at least in part. This is illustrated on Fig. 8, tube *D*, which was heated to 440 C. for 1 hr., after plating, but prior to cutting of the tongue. In the figure, a $^3/_{16}$-in. ball is resting on the free end of the tongue, which is depressed below the surface of the tube. The tongue deflected so that the chromium was on its convex surface. This indicated that the chromium had been permanently elongated during the heat treatment due to expansion of the steel; there had been either plastic flow or fractures in the chromium or both.

On the basis of the foregoing discussion, it is postulated that the residual tensile stresses in electrodeposited chromium are a major factor in adversely affecting the endurance limits of steel upon which the chromium is plated. Heating at temperatures of 100 to 300 C. tends to contract the chromium; the underlying steel resists this contraction, thus producing increased tensile stresses in the chromium and further reducing the endurance limit. Contraction of the chromium during heating at 400 and 440 C., coupled with the expansion of the steel at these temperatures, stressed the chromium beyond its ultimate tensile strength, produced permanent deformation, and hence relieved the stresses in the chromium to some extent with resulting increases in endurance limits of the plated specimens so treated.

Summary

1. Chromium plating of S.A.E. X4130 and 6130 steels reduced the endurance limits of these materials, as determined in the rotating-beam type of test.

2. The endurance limits of these steels plated under a given set of conditions generally decreased with increased hardness of the steel. For a steel of a given hardness (Rockwell 40 C), the reductions in endurance limit increased with increased plating bath temperatures.

3. The endurance limits of specimens plated, and subsequently surface ground, were equal to or greater than those of unground specimens having the same final plating thickness. Interruption of the plating process did not affect the endurance limit of the plated specimens, provided that proper precautions for plating chromium on chromium were taken before the plating was resumed.

4. The endurance limits of specimens heated after plating and tested at room temperature reached a minimum value for a baking temperature between 190 and 300 C. Endurance limits of specimens heated for 1 hr. at 440 C. were significantly higher than those of the "as-plated" steel.

5. Reduction of the endurance limits of steel by chromium plating is believed to be due, in part at least, to residual tensile stresses in the chromium. Heating of chromium increased its density and hence decreased its volume. It is postulated that heating at low temperatures increased the residual tensile stresses in the chromium; at sufficiently high temperatures, the concurrent contraction of the chromium and expansion of the steel stressed the chromium above its ultimate tensile strength, thus relieving some of the residual tensile stresses in the chromium and increasing the endurance limit of the plated specimens.

Acknowledgment:

The author gratefully acknowledges the valued advice, aid, and assistance of the following in various phases of this investigation: Naval Aircraft Factory, Philadelphia; N. E. Promisel, Chief Metallurgist, Bureau of Aeronautics, Navy Department, Washington; William Blum and Vernon Lamb of the Bureau's Electrodeposition Section, Harold Hessing, Houston Babb, Thomas P. Royston, Jr., Irene C. Minor, and Lura F. Roehl of the Metallurgy Division of the Bureau. Harold E. Francis, Guy R. McConnell, Richard C. Rohan, and Molly Thompson, formerly of the metallurgy Division, also assisted in the investigation.

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DISCUSSION

MR. L. R. JACKSON (*presented in written form*).[1]—The author is to be congratulated on his careful handling of an intricate experimental problem.

The fact that chromium plate usually has an adverse effect on the fatigue strength of plated steel parts has long been known. There has been confusion, however, regarding the mechanism by which this adverse effect is achieved. The present paper goes far toward reducing that confusion.

From this paper, the three mechanisms, arranged in the order of their importance, by which chromium plate reduces the fatigue strength of steel, now appear to be:

1. Unfavorable residual stresses.
2. Cracks in the chromium plate.
3. Hydrogen embrittlement.

Furthermore, as Mr. Logan demonstrated, the third of these mechanisms is, at most, of minor importance.

In view of the fact that it is often desirable to chromium-plate parts that will be subjected to repeated stress, Mr. Logan's results should be of value in guiding research on chromium plating in useful channels.

MR. GEORGE DUBPERNELL (*presented in written form*).[2]—In connection with the Bureau of Standards' work, it may be of interest to call attention to the development of a special type of chromium plate which can be heat treated without cracking, and which has a minimum effect on the fatigue strength of steel. This process found application on the chromium plating of propeller blades at the Curtiss-Wright plant at Caldwell, N. J. The process of producing this special type of chromium plate has been disclosed in U. S. Patent No. 2,462,615, February 22, 1949. It has also been disclosed to some extent in several published discussions by S. Weisman.[3]

MR. R. L. TEMPLIN.[4]—The author has presented one of the typical messes that the fatigue investigator gets into. We have done a little work with chromium plating on aluminum, and I was sorry that he failed to mention some work, I believe, done at the National Bureau of Standards in attempting to evaluate the modulus of elasticity of chromium plating and reveal the absence of ductility of such plating. If a metal having a high modulus is plated on one that has a low modulus and the plating has a very low ductility, I think it would be reasonable to expect that under suitable stressing the plating would crack. And if the cracks occur, the assumption is that they would be reasonable causes of early fatigue failure, because of stress concentrations.

The N.B.S. investigation referred to resulted in the author's suggesting that a reliable value for Young's modulus of elasticity for good chromium plate was in the neighborhood of 36,000,000 psi., although the highest experimental value they obtained was about 33,000,000 psi. Some experimental work done at the Aluminum Research Laboratories gave a value of 32,800,000 psi. which is considerably higher than Young's modulus for aluminum. In the Aluminum Research Laboratory tests the chromium plating cracked when the tensile stress in the chromium-plated aluminum specimen reached a value of about 28,000 to 29,000 psi., or at a strain value of 0.0026 in. per in. These cracks, in the case of fatigue specimens, apparently act as stress raisers and have a deleterious effect on the fatigue strength of the material.

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[2] Manager, Waterbury Electroplating Lab., United Chromium, Inc., Waterbury, Conn.

[3] *Transactions*, Electrochemical Soc., Vol. 86, pp. 454–455 (1944) and *Proceedings*, Thirty-sixth Convention, Am. Electroplaters' Soc., pp. 136–137 (1949).

[4] Assistant Director of Research and Chief Engineer of Tests, Research Laboratories, Aluminum Company of America, New Kensington, Pa.

The problem presented is of particular interest in chromium-plated steel dies which in use may be expected to withstand stresses and strains beyond those which can be withstood by the plating. Consequently the plating cracks and separates from the steel. Often an analysis of the problem presented results in the conclusion that the coating failed to adhere properly, but in view of the strength and deformation limits just mentioned it is difficult to understand how the plating could be expected to adhere when the stresses and strains are in excess of those the plating will withstand.

MR. G. R. GOHN.[5]—In 1937, Mr. Greenall and I plated both chromium and nickel separately and nickel and chromium together on some phosphor bronze strip specimens and subjected them to bending fatigue stresses. We observed that there were residual tensile stresses in the "as-plated" material which materially reduced the fatigue strength of the plated parts. However, when compressive stresses were put in the specimens by polishing, prior to plating, the normal fatigue strength of the unplated material was obtained in test. Further improvement was obtained if, after plating, additional compressive stresses were imposed by buffing or polishing the plated material. These data may be found in the 1937 *Proceedings*.[6]

MR. H. L. LOGAN (*author's closure*).—I wish to thank those who took part in the discussion for their contribution to the paper.

The work at the National Bureau of Standards on Young's modulus of elasticity of plated chromium, discussed by Mr. Templin, was done by Mr. Brenner[7] and his co-workers. They reported values ranging from 12×10^6 to 33×10^6 psi. The higher values were obtained on specimens plated in baths maintained at 85 C. or above.

Cracking of the plating such as Mr. Templin postulates would unquestionably lead to early fatigue failures. However, results of an investigation to determine whether fatigue cracks originated in the chromium or steel failed to reveal cracking to which we could attribute the fatigue failures. Applied stresses in that case were slightly above the fatigue limit obtained for similar specimens in the present investigation. A strain of 0.0026 in. per in., which Mr. Templin stated caused cracking in chromium plated on an aluminum alloy would of course correspond to stresses in steel 2.6 to 2.9 times the values of 28,000 to 29,000 psi. which Mr. Templin reported. Stresses of that magnitude were well above any endurance strengths which we obtained for the unbaked specimens.

In an investigation of the effect of

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[5] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[6] C. H. Greenall and G. R. Gohn, "Fatigue Properties of Non-Ferrous Sheet Metals," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, Part II, p. 160 (1937).

[7] A. Brenner, P. Burkhead, and C. Jennings, Physical Properties of Electrodeposited Chromium," *Journal of Research*, National Bureau of Standards, Vol. 40, p. 31 (1948). (*RP* 1854).

chromium plating on the plastic deformation of S.A.E. 4130 steel[8] we found no separation of the plating from the steel in the elastic range. The material tested had a yield strength (0.2 per cent offset from modulus line) of about 175,000 psi.

Mr. Gohn's observations appear logical since we have found that the fatigue limit of specimens, in general, was increased by grinding after plating.

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[8] H. L. Logan, "Effect of Chromium Plating on the Plastic Deformation of S.A.E. 4130 Steel," to be published in *Journal of Research*, Nat. Bureau of Standards about June, 1951.

FATIGUE NOTCH-SENSITIVITIES OF SOME AIRCRAFT MATERIALS*

By Horace J. Grover[1]

Most service fatigue failures of structural parts occur at notches, grooves, holes, fillets, or other sharp changes in geometrical section. These failures usually occur in lifetimes far shorter than would be expected for the material concerned, under the nominal stresses imposed. Special allowance for the effects of necessary notches may thus be of considerable importance in the design of structures to withstand repeated stresses. Design allowance for notches is of particular interest in aircraft structures where notched parts are expected to undergo varied stresses and yet weight penalties for over-design are serious.

In the course of a partly completed survey, at Battelle, of available information on the fatigue properties of structural materials, there has been occasion to review data on fatigue notch sensitivities of a number of materials used in aircraft. This paper presents the results of this review in a form that permits some appraisal of the nature and extent of information available to the designer.

Collected Experimental Data

Tables I through IV show data collected from available reports of notch-fatigue tests on the following materials: three S.A.E. steels (4130, 4140, and 4340), two aluminum alloys (24S-T and 75S-T), and 18-8 stainless steel.

For convenience in later discussion, these data have been arranged according to the type of repeated loading:

Table I.—Rotating-beam test results.

Table II.—Repeated-bending test results.

Table III.—Repeated-torsion test results.

Table IV.—Axial-loading test results.

All data available for the first three tables are for tests in which outer fibers of specimens were subjected to fully reversed nominal stresses (although this is not a necessary limitation in plate bending or repeated torsion). Results of axial-loading fatigue tests include several instances in which the mean nominal stress differs from zero.

Within each table, data from various sources have been arranged in a form suitable for some intercomparison. In several instances this has required slight interpolation or extrapolation of available results; moreover, compact summary has necessitated omission of some experimental details. For more complete account of specific items, reference should be made to original reports and papers.

Before attempting appraisal of the whole body of data in these tables, it is desirable to examine separately the information available from each type of test.

Effects of Notches on Rotating-Beam Fatigue Strengths

In Table I (Tables II, III, and IV as well) there is listed, for each notch form, a value K_T. This is the "theoretical stress-concentration factor" and designates, for an ideal, homogeneous, isotropic, and completely elastic material,[2] the ratio of maximum local stress to nominal stress.

One can define a "fatigue-strength re-

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* This paper was presented at the Session on Fatigue of Metals held at the First Pacific Area National Meeting of the Society, San Francisco, Calif., October 10–14, 1949.

[1] Battelle Memorial Inst., Columbus, Ohio.

[2] Values of K_T listed in the tables are either those quoted in original references or values obtained (from quoted notch dimensions) by the nomograph for α_K in Neuber's book (**11**).

TABLE I.—ROTATING-BEAM NOTCH-FATIGUE STRENGTHS.

	Condition	Mechanical-Strength Properties				Diameter of Specimen, in.	Notch Specifications		Fatigue Strengths at Indicated Numbers of Cycles to Failure, psi.						Reference Number[b]
		Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Reduction of Area, per cent		Type of Notch[a]	K_T	50 000	100 000	500 000	1 000 000	5 000 000	10 000 000	
						S.A.E. 4130[c, d, e]									
No. 1	Normalized	76 200	45 000	32	66	0.250	Unnotched		...	48 000	45 000	44 000	42 500	42 500	1
		76 200	45 000	32	66	0.250	A	2.6	...	....	30 000	26 000	25 000	25 000	1
No. 2	Oil quenched, drawn 1100 F.	139 500	120 000	18	63	0.250	Unnotched		...	...	91 000	88 000	86 000	86 000	1
		139 500	120 000	18	63	0.250	A	2.6	...	...	37 500	35 000	30 000	30 000	1
No. 3	Oil quenched, drawn 650 F.	199 300	168 500	10	45	0.250	Unnotched		...	...	118 000	116 000	114 000	114 000	1
		199 300	168 500	10	45	0.250	A	2.6	...	...	58 000	55 000	53 000	53 000	1
No. 4	Quenched and drawn	141 800	115 600	18	60	0.125	Unnotched		...	...	81 000	78 000	76 000	75 400	2
		141 800	115 600	18	60	0.125	B	2.0	...	...	41 000	40 000	39 500	38 600	2
No. 5		141 800	115 600	18	60	0.250	Unnotched		...	...	79 000	74 000	70 000	69 800	2
		141 800	115 600	18	60	0.250	C	2.0	...	57 000	45 000	42 000	39 000	37 300	2
No. 6		141 800	115 600	18	60	0.500	Unnotched		...	...	69 000	68 000	66 000	65 000	2
		141 800	115 600	18	60	0.500	D	2.0	...	...	43 000	39 000	37 500	35 800	2
No. 7		141 800	115 600	18	60	1.000	Unnotched		...	...	66 000	64 000	64 000	63 600	2
		141 800	115 600	18	60	1.000	E	2.0	...	...	39 000	37 000	35 000	34 600	2
No. 8		141 800	115 600	18	60	1.750	Unnotched		...	80 000	67 000	65 000	64 000	63 600	2
		141 800	115 600	18	60	1.750	F	2.06	...	...	...	39 000	35 000	34 800	2
No. 9	Normalized drawn 850 F.	104 300	80 500	23	63	0.230	Unnotched		66 000	62 000	52 000	47 500	47 000	47 000	3
		104 300	80 500	23	63	0.230	G	2.9	...	42 000	33 000	28 500	23 000	23 000	3
No. 10	Oil quenched, drawn 1010 F.	151 500	144 200	16	63	0.230	Unnotched		101 000	95 000	84 000	80 000	77 500	77 500	3
		151 500	144 200	16	63	0.230	G	2.9	51 000	47 000	38 000	33 000	28 500	28 500	3
No. 11	Oil quenched, drawn 850 F.	185 800	173 800	11	55	0.230	Unnotched		114 000	103 000	88 000	88 000	87 500	87 500	3
		185 800	173 800	11	55	0.230	G	2.9	60 000	55 000	42 000	38 500	38 500	38 500	3
						S.A.E. 4140									
No. 12	Oil quenched, drawn 1200 F.	133 000	113 000	19	66	0.215	Unnotched		98 000	92 000	82 000	80 000	79 000	79 000	4
		133 000	113 000	19	66	0.215	H	3.1	50 000	44 000	34 000	31 000	30 000	30 000	4
		133 000	113 000	19	66	0.215	I	2.1	55 000	49 000	39 000	38 000	38 000	38 000	4
		133 000	113 000	19	66	0.215	J	1.6	63 000	55 000	49 000	49 000	49 000	49 000	4
No. 13	Oil quenched, drawn 450 F.	275 500	222 500	11	48	0.215	Unnotched		171 000	152 000	122 000	119 000	117 000	117 000	4
		275 500	222 500	11	48	0.215	H	3.1	72 000	65 000	60 000	60 000	60 000	60 000	4
		275 500	222 500	11	48	0.215	I	2.1	93 000	86 000	77 000	76 000	75 000	75 000	4
		275 500	222 500	11	48	0.215	J	1.6	107 000	100 000	100 000	100 000	100 000	100 000	4

						S.A.E. 4340[f]									
No. 14	Oil quenched drawn 1000 F.	163 700	...	16	56	0.125	Unnotched		112 500	102 500	84 000	83 000	82 500	82 500	5
		163 700	...	16	56	0.125	B	2.0	72 000	65 000	52 000	51 700	51 700	51 700	5
No. 15		163 700	...	16	56	0.250	Unnotched		110 000	98 000	81 000	81 000	81 000	81 000	5
		163 700	...	16	56	0.250	C	2.0	69 500	62 500	49 000	48 000	48 000	48 000	5
No. 16		163 700	...	16	56	0.500	Unnotched		104 000	93 000	79 000	78 000	78 000	78 000	5
		163 700	...	16	56	0.500	D	2.0	66 000	60 000	49 000	48 000	48 000	48 000	5
No. 17		163 700	...	16	56	1.000	Unnotched		104 000	93 000	74 000	74 000	74 000	74 000	5
		163 700	...	16	56	1.000	E	2.00	65 000	58 000	46 000	46 000	46 000	46 000	5
No. 18		1 750	...	16	56	1.750	Unnotched		...	97 000	74 000	74 000	74 000	74 000	5
		163 700	...	16	56	1.750	K	2.0	63 000	57 000	42 000	42 000	42 000	42 000	5
						24 S-T									
No. 19	Extrusion	80 300	62 900	...	...	0.250	Unnotched		...	35 500	...	...	...	29 500	1
		80 300	62 900	22	...	0.250	A	2.6	...	...	...	...	18 800	12 500	1
No. 20	Extrusion	68 000	46 000	22	...	0.300	Unnotched		...	45 000	...	...		24 000	6
		68 000	46 000	22	...	0.295	L	2.0	...	25 000	...	...	...	13 000	6
		68 000	46 000	22	...	0.300	M	5.0	...	23 000	...	...	...	10 500	6
						75 S-T[g]									
No. 21	...	86 200	76 000	9	15	0.125	Unnotched		52 000	43 500	39 000	36 100	34 400	32 600	7
	...	86 200	76 000	9	15	0.125	B	2.0	25 000	19 800	16 000	15 800	15 000	14 100	7
No. 22	...	86 200	76 000	9	15	0.250	Unnotched		49 600	45 200	38 500	37 000	34 500	32 000	7
	...	86 200	76 000	9	15	0.250	C	2.0	30 500	27 000	21 000	20 000	18 500	17 000	7
No. 23	...	86 200	76 000	9	15	0.500	Unnotched		49 000	44 000	37 000	35 000	32 500	29 400	7
	...	86 200	76 000	9	15	0.500	D	2.0	27 000	25 200	22 000	20 800	18 600	16 400	7
No. 24	...	86 200	76 000	9	15	1.000	Unnotched		51 700	47 000	36 000	32 000	29 600	27 100	7
	...	86 200	76 000	9	15	1.000	E	2.0	34 000	31 000	24 200	21 700	18 400	15 100	7
No. 25	...	86 200	76 000	9	15	1.750	Unnotched		48 200	42 000	34 000	31 800	28 800	25 600	7
	...	86 200	76 000	9	15	1.750	K	2.0	30 800	27 500	22 300	20 800	19 000	17 000	7
						18-8 STAINLESS									
No. 26	Annealed	82 800	34 800	64	76	0.250	Unnotched		...	...	38 000	35 000	35 000	35 000	1
		82 800	34 800	64	76	0.250	A	2.6	...	45 000	44 000	43 000	43 000	43 000	1
No. 27	Cold drawn	132 500	109 000	32	72	0.250	Unnotched		...	78 000	71 000	70 000	70 000	70 000	1
		132 500	109 000	32	72	0.250	A	2.6	...	...	53 000	49 000	49 000	49 000	1

(Footnotes on page 720)

TABLE I.—FOOTNOTES.

[a] *Details of Notches:*

Notch	Type	Diameter of bar, in.	Diameter at Root Notch, in.	Notch Radius, in.	K_T
A	60-deg. V groove	0.300	0.250	0.010	2.6
B	Semi-circular	0.145	0.125	0.010	2.0
C	Semi-circular	0.290	0.250	0.020	2.0
D	Semi-circular	0.580	0.500	0.040	2.0
E	Semi-circular	1.160	1.000	0.080	2.0
F	Semi-circular	2.030	1.750	0.140	2.1
G	60-deg. V groove	0.400	0.230	0.010	2.9
H	60-deg. V groove	0.400	0.215	0.010	3.1
I	60-deg. V groove	0.400	0.215	0.025	2.1
J	60-deg. V groove	0.400	0.215	0.060	1.6
K	Semi-circular	2.175	1.875	0.150	2.0
L	Semi-circular	0.354	0.295	0.0295	2.0
M	60-deg. V groove	0.480	0.300	0.002	5.0

[b] These numbers refer to the list of references appended to this paper, see p. 729.

[c] Values quoted for Nos. 1, 2, and 3 are averages for the two testing speeds (see reference); in general, results for the two speeds did not differ greatly.

[d] Values quoted in Nos. 4 to 8 are for a single heat of steel from a study of size effect. Tests were made on several rotating-cantilever-beam-type machines.

[e] Values quoted in Nos. 9 to 11 are for a single heat of steel, heat treated to different hardnesses.

[f] Values quoted for Nos. 14 to 18 are for a single heat of steel from a study of size effects. Tests were made on several rotating-cantilever-beam-type machines.

[g] Values quoted in Nos. 21 to 25 are from a study of size effect on 75 S-T. Tests were run on rotating-cantilever beam machines.

TABLE II.—REPEATED BENDING NOTCH-FATIGUE STRENGTHS OF 24S-T ALUMINUM ALLOY.

	Condition	Mechanical-Strength Properties				Specimen Width, in.	Notch Specifications		Fatigue Strengths at Indicated Numbers of Cycles						Reference Number[a]
		Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Reduction of Area, per cent		Type of Notch	K_T	50 000	100 000	500 000	1 000 000	5 000 000	10 000 000	
No. 1	0.020-in. sheet	68 000	46 000	19	...	0.650	Unnotched		...	43 000	...	...	...	25 500	6
		68 000	46 000	19	...	0.525	Edge	1.6	...	37 000	...	...	...	19 000	6
No. 2	0.064-in. sheet	68 000	46 000	19	...	0.650	Unnotched		...	45 000	...	...	...	22 500	6
		68 000	46 000	19	...	0.650	Surface-notch	2.0	...	32 500	...	...	...	19 000	6

[a] These numbers refer to the list of references appended to this paper, see p. 729.

duction factor" for a particular specimen tested in rotating bending as

$$K_f = \frac{\text{fatigue strength of unnotched specimen at } N \text{ cycles}}{\text{fatigue strength of notched specimen at } N \text{ cycles}}$$

TABLE III.—TORSION NOTCH-FATIGUE STRENGTHS OF 75S-T ALUMINUM ALLOY.

NOTE.—Torsion Modulus Rupture 57,000 psi. Torsion Yield Strength 44,700 psi.

Mechanical-Strength Properties				Specimen Size, in.	Notch Specifications		Fatigue Strengths Indicated Numbers of Cycles						Reference Number[a]
Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Reduction of Area, per cent		Type of Notch	K_T	50 000	100 000	500 000	1 000 000	5 000 000	10 000 000	
86.2	76	9	16	1/4	Unnotched		32 500	30 000	24 000	22 000	19 000	19 000	8
86.2	76	9	16	1/4	Semi-circular groove (ρ = 0.02 in.)	1.5	25 000	22 500	17 500	15 500	12 500	**12 000**	8

[a] These numbers refer to the test of references appended to this paper, see p. 729.

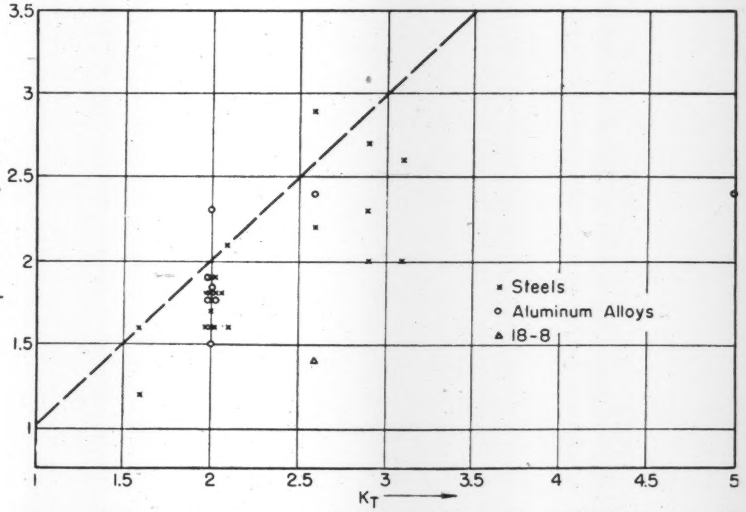

FIG. 1.—Variation of K_f (Fatigue-Strength Reduction Factor at 10^7 Cycles) with K_T (Stress Concentration Factor) for Rotating-Beam Test Data Listed in Table I.

It should be emphasized that K_f, so defined, may vary with lifetime as well as with type of notch even for specimens of one material under one type of repeated loading.

Figure 1 shows K_f at 10^7 cycles plotted against K_T for all items listed in Table I; Fig. 2 shows K_f at 10^5 cycles for all items for which values at this lifetime are listed. Several observations may be drawn from these figures:

1. K_f is usually less than K_T. (Actually, the few cases in which K_f appears slightly greater than K_T are believed to be ascribable to experimental error.)
2. K_f/K_T seems to decrease as K_T increases, although not many values are available for high K_T.

TABLE IV.—AXIAL-LOADING NOTCH-FATIGUE STRENGTHS.

	Condition	Mechanical-Strength Properties				Specimen Width, in.	Notch Specifications		Loading Condition	Fatigue Strengths at Indicated Numbers of Cycles, psi.						Reference Number[a]
		Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Reduction in Area, per cent		Type	K_T		50 000	100 000	500 000	1 000 000	5 000 000	10 000 000	
							24 S-T									
No. 1.....	0.032-in. sheet	71 500	51 600	18	...	¼	Unnotched		Fully reversed stress	37 000	32 500	26 500	24 500	22 500	22 000	8, 9
	0.032-in. sheet	71 500	51 600	18	...	¼	0.02-in. hole	2.8	Fully reversed stress	27 500	25 000	20 000	18 000	17 000	16 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	¼	1/16-in. hole	2.4	Fully reversed stress	29 000	23 000	17 000	15 500	13 000	12 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	¼	⅛-in. hole	2.2	Fully reversed stress	25 000	22 500	19 000	18 000	17 500	17 500	8, 9
No. 2.....	0.032-in. sheet	71 500	51 600	18	...	½	Unnotched		Fully reversed stress	40 000	35 000	27 000	25 000	22 500	22 000	8, 9
	0.032-in. sheet	71 500	51 600	18	...	½	⅛-in. hole	2.4	Fully reversed stress	22 500	20 000	17 000	15 000	13 000	12 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	½	¼-in. hole	2.2	Fully reversed stress	21 500	19 000	15 000	13 000	12 500	12 000	8, 9
	0.032-in. sheet	71 500	51 600	18	...	½	⅜-in. hole	2.0	Fully reversed stress	22 000	19 000	15 000	14 000	12 500	12 000	8, 9
No. 3.....	0.030-in. sheet	71 500	51 600	18	...	1	Unnotched		Fully reversed stress	34 500	30 500	24 000	22 500	20 000	19 000	8, 9
	0.030-in. sheet	71 500	51 600	18	...	1	¼-in. hole	2.4	Fully reversed stress	20 000	17 500	13 000	12 000	11 500	11 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	1	½-in. hole	2.2	Fully reversed stress	21 500	19 000	15 000	13 000	12 500	12 000	8, 9
	0.032-in. sheet	71 500	51 600	18	...	1	¾-in. hole	2.0	Fully reversed stress	22 000	20 000	17 000	15 000	13 000	12 500	8, 9
No. 4.....	0.032-in. sheet	71 500	51 600	18	...	2	Unnotched		Fully reversed stress	...	...	...	23 000	23 000	22 000	8, 9
	0.032-in. sheet	71 500	51 600	18	...	2	0.02-in. hole	2.9	Fully reversed stress	...	23 000	18 500	17 500	15 000	14 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	2	½-in. hole	2.4	Fully reversed stress	18 500	17 000	13 000	12 000	11 500	11 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	2	1-in. hole	2.2	Fully reversed stress	20 000	17 500	13 000	12 000	10 500	10 500	8, 9
	0.032-in. sheet	71 500	51 600	18	...	2	⅜-in. hole	2.0	Fully reversed stress	18 000	16 500	13 000	12 500	12 000	12 000	8, 9
No. 5.....	0.064-in. sheet	71 500	51 600	18	...	¼	Unnotched		Fully reversed stress	36 000	32 000	24 000	22 500	22 000	21 500	8, 9
	0.064-in. sheet	71 500	51 600	18	...	¼	0.02-in. hole	2.8	Fully reversed stress	27 500	25 000	20 000	18 500	16 000	15 000	8, 9
	0.064-in. sheet	71 500	51 600	18	...	¼	1/16-in. hole	2.4	Fully reversed stress	...	20 500	16 500	15 000	14 000	13 500	8, 9
	0.064-in. sheet	71 500	51 600	18	...	¼	⅛-in. hole	2.2	Fully reversed stress	23 500	22 000	17 500	16 500	15 000	15 000	8, 9
	0.064-in. sheet	71 500	51 600	18	...	¼	3/16-in. hole	2.0	Fully reversed stress	25 000	22 500	18 500	18 000	17 500	17 500	8, 9
N . 6.....	0.064-in. sheet	71 500	51 600	18	...	½	Unnotched		Fully reversed stress	...	32 000	23 500	22 000	20 000	20 000	8, 9
	0.064-in. sheet	71 500	51 600	18	...	½	⅛-in. hole	2.4	Fully reversed stress	20 500	18 500	14 500	13 500	12 500	12 000	8 9
	0.064-in. sheet	71 500	51 600	18	...	½	¼-in. hole	2.2	Fully reversed stress	22 500	20 000	16 000	15 000	14 500	14 000	8, 9
	0.064-in. sheet	71 500	51 600	18	...	½	⅜-in. hole	2.0	Fully reversed stress	23 000	20 500	16 500	15 000	14 500	14 000	8, 9
No. 7.....	0.064-in. sheet	71 500	51 600	18	...	1	Unnotched		Fully reversed stress	...	...	23 000	21 500	18 500	18 000	8, 9
	0.064-in. sheet	71 500	51 600	18	...	1	¼-in. hole	2.4	Fully reversed stress	...	16 000	12 500	11 500	11 500	10 500	8, 9
	0.064-in. sheet	71 500	51 600	18	...	1	½-in. hole	2.2	Fully reversed stress	19 500	17 500	13 000	12 000	11 500	11 500	8, 9
	0.064-in. sheet	71 500	51 600	18	...	1	¾-in. hole	2.0	Fully reversed stress	21 500	18 500	14 000	12 500	11 000	10 500	9, 9
No. 8.....	0.064-in. sheet	71 500	51 600	18	...	2	½-in. hole	2.4	Fully reversed stress	...	...	14 000	13 000	12 500	11 500	8, 9
	0.064-in. sheet	71 500	51 600	18	...	2	¼-in. hole	2.2	Fully reversed stress	...	18 000	14 000	12 500	12 000	11 500	8, 9
	0.064-in. sheet	71 500	51 600	18	...	2	1½-in. hole	2.0	Fully reversed stress	21 500	18 500	15 000	14 000	13 500	13 000	8, 9
	0.064-in. sheet	71 500	51 600	18	...	2	⅙-in. hole	1.9	Fully reversed stress	23 000	20 500	16 500	15 500	14 500	14 000	8, 9
No. 9.....	0.040-in. sheet	73 700	54 000	16	...	1	Unnotched		$R = +0.60$	...	70 500	70 000	69 500	69 200	69 000	10
	0.040-in. sheet	73 700	54 000	16	...	1.5	0.375-in. hole	2.6	$R = +0.60$	51 000	47 000	42 000	41 000	40 500	40 000	10
No. 10.....	0.040-in. sheet	73 700	54 000	16	...	1	Unnotched		$R = +0.50$	...	(68 500	67 500	66 500	66 200	66 000)[b]	10
	0.040-in. sheet	73 700	54 000	16	...	1.5	0.375-in. hole	2.6	$R = +0.50$	48 000	44 000	37 000	36 500	36 200	36 000	10

No. 11	0.040-in. sheet	73 700	54 000	16	...	1	Unnotched		$R = +0.40$	70 500	67 000	63 000	62 500	62 100	62 000	10
	0.040-in. sheet	73 700	54 000	16	...	1.5	0.375-in. hole	2.6	$R = +0.40$	43 000	39 000	31 500	31 000	30 700	35 000	10
No. 12	0.040-in. sheet	73 700	54 000	16	...	1	Unnotched		$R = +0.25$	65 000	60 000	53 000	52 000	52 000	52 000	10
	0.040-in. sheet	73 700	54 000	16	...	1.5	0.375-in. hole	2.6	$R = +0.25$	35 000	33 000	28 000	26 000	25 500	25 000	10
No. 13	0.064-in. sheet	68 000	46 000	19	...	......	Unnotched		$R = +0.25$	...	58 000	...	40 000	...	27 000	6
	0.064-in. sheet	68 000	46 000	19	...	......	Surface grooves	5.0	$R = +0.25$	...	45 000	...	37 500	...	34 000	6
	0.064-in. sheet	68 000	46 000	19	...	......	0.1 in. hole	2.6	$R = +0.25$	...	32 000	...	23 500	...	23 000	6
24 S-T ALCLAD																
No. 14	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = +0.75$	67 500	67 000	65 000	63 000	62 000	61 000	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = +0.75$	...	55 000	40 000	34 000	28 000	25 000	10
No. 15	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = +0.60$	...	63 000	51 000	47 500	43 000	40 500	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = +0.60$	45 000	42 000	30 500	27 000	23 000	22 000	10
No. 16	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = +0.40$	63 000	56 000	40 000	36 000	31 000	29 000	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = +0.40$	40 000	36 000	26 000	23 000	(19 000 | 17 500)[b]	10
No. 17	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = +0.25$	56 000	49 000	35 000	31 500	27 000	26 000	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = +0.25$	35 000	29 500	19 000	16 500	14 000	13 000	10
No. 18	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = +0.02$	45 000	40 000	27 000	24 000	22 000	21 000	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = +0.02$	28 000	25 000	16 000	(14 000	12 500	11 500)[b]	10
No. 19	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = -0.25$	(42 500	38 000	24 500	20 000	29 000	18 000)[b]	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = -0.25$	25 000	21 500	21 000	13 000	(11 000 | 10 500)[b]	10
No. 20	0.040-in. sheet	69 400	51 900	15	...	1	Unnotched		$R = -0.50$	40 000	36 000	22 000	19 000	17 000	16 000	10
	0.040-in. sheet	69 400	51 900	15	...	1.5	0.375-in. hole	2.6	$R = -0.50$	21 000	15 500	15 500	10 000	9 200	9 000	10
75 S-T																
No. 21	0.032-in. sheet	85 000	77 400	10	...	¼	Unnotched		$R = -1$	43 000	38 000	28 500	26 000	23 500	23 000	8
	0.032-in. sheet	85 000	77 400	10	...	¼	0.02-inch hole	2.8	$R = -1$	27 500	25 000	20 000	18 500	17 000	16 500	8
	0.032-in. sheet	85 000	77 400	10	...	¼	1/16-inch hole	2.4	$R = -1$	23 500	21 000	15 000	13 000	12 000	11 500	8
	0.032-in. sheet	85 000	77 400	10	...	¼	⅛-inch hole	2.2	$R = -1$	24 500	22 000	15 500	14 000	12 000	11 500	8
No. 22	0.032-in. sheet	85 000	77 400	10	...	½	Unnotched		$R = -1$	40 500	35 500	26 500	22 500	18 000	17 500	8
	0.032-in. sheet	85 000	77 400	10	...	½	⅛-inch hole	2.4	$R = -1$	20 000	17 000	12 000	11 000	10 500	10 000	8
	0.032-in. sheet	85 000	77 400	10	...	½	¼-inch hole	2.2	$R = -1$	23 500	31 500	15 500	14 000	12 000	11 500	8
	0.032-in. sheet	85 000	77 400	10	...	½	⅜-inch hole	2.0	$R = -1$	23 000	20 000	15 000	13 000	10 500	10 000	8
No. 23	0.032-in. sheet	85 000	77 400	10	...	1	Unnotched		$R = -1$	33 500	30 500	24 500	23 000	19 500	19 000	8
	0.032-in. sheet	85 000	77 400	10	...	1	¼-inch hole	2.4	$R = -1$	20 000	17 000	12 000	11 000	10 500	10 000	8
	0.032-in. sheet	85 000	77 400	10	...	1	½-inch hole	2.2	$R = -1$	12 000	18 000	13 000	12 000	10 500	10 000	8
	0.032-in. sheet	85 000	77 400	10	...	1	¾-inch hole	2.0	$R = -1$	23 000	20 000	15 000	13 000	10 500	10 000	8
No. 24	0.032-in. sheet	85 000	77 400	10	...	2	Unnotched		$R = -1$	...	...	21 500	19 500	16 500	15 500	8
	0.032-in. sheet	85 000	77 400	10	...	2	0.02-in. hole	2.9	$R = -1$	...	...	19 000	17 500	15 000	14 500	8
	0.032-in. sheet	85 000	77 400	10	...	2	½-in. hole	2.4	$R = -1$	20 000	17 000	12 000	11 000	10 500	10 000	8
	0.032-in. sheet	85 000	77 400	10	...	2	1-in. hole	2.2	$R = -1$	22 000	18 000	13 000	12 000	10 500	10 000	8
	0.032-in. sheet	85 000	77 400	10	...	2	1½-in. hole	2.0	$R = -1$	21 000	18 000	13 000	12 000	10 500	10 000	8
TYPE 301 (STAINLESS)																
No. 25	(0.020-in. sheet	169 000	125 000	33	...	1	Unnotched		$R = +0.25$	150 000	146 000	135 000	130 000	125 000	122 000	4
	½ hard)	169 000	125 000	33	...	1	⅛-in. hole	2.6	$R = +0.25$	74 000	70 000	60 000	57 000	48 000	45 000	4

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[a] These numbers refer to the list of references appended to this paper, see p. 729.

[b] By interpolation.

3. K_f/K_T is generally larger for the larger value of N (compare Figs. 1 and 2).

In the partly completed survey of additional structural materials, these observations appear also valid for other alloys. It thus seems that using unnotched fatigue strength values[3] and theoretical stress-concentration factors will generally lead to conservative design for members subject to reversed bending of the type present in these rotating-beam tests.

Further analysis of the data shown in Fig. 1 (and of available data on other materials) has not shown systematic trends. Thus, while one group of test results shows, for a particular notch, an apparent relation between K_f (at fixed lifetime) and the strength level of a steel or between K_f and ductility indicated by a yield ratio, such relations have not been found applicable to accumulated data from many sources. Factors that may be responsible for some variations will be discussed later. For the present, the data in Table I do not afford any apparent simple measure of fatigue notch sensitivities of the materials concerned.

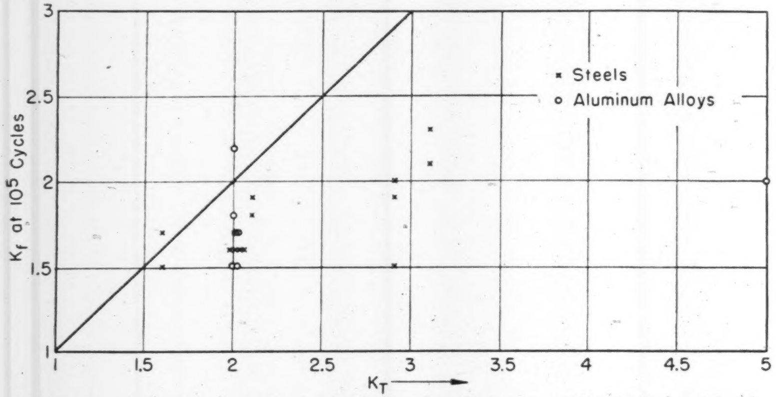

FIG. 2.—Variation of K_f (Fatigue-Strength Reduction Factor at 10^5 Cycles) with K_T (Stress Concentration Factor)for Rotating-Beam Test Data Listed in Table I).

TABLE V.—ILLUSTRATION OF COMPUTATION OF FATIGUE-STRENGTH REDUCTION FACTORS FOR A PARTICULAR CASE (NO. 12, TABLE IV).

Specimen	Nominal Stress Values for Lifetime of 10^7 Cycles				
	Load Ratio, R	Maximum Stress, psi.	Minimum Stress, psi.	Mean Stress, psi.	Stress Amplitude, psi.
Notched	+0.40	35 000	14 000	24 500	10 500
Unnotched	+0.40	62 000	24 800	43 400	18 600
Unnotched[a]	+0.13	44 000	5 000	24 500	19 500

$$K_f' = \frac{\textit{Stress amplitude unnotched specimen}}{\textit{Stress amplitude notched specimen at same mean stress}} = \frac{19.5}{10.6} = 1.84$$

$$K_f'' = \frac{\textit{Maximum stress unnotched specimen}}{\textit{Maximum stress notched specimen at same } R} = \frac{62}{35} = 1.77$$

$$K_f''' = \frac{\textit{Maximum stress unnotched specimen}}{\textit{Maximum stress notched specimen at same mean stress}} = \frac{44}{35} = 1.26$$

[a] Values from interpolation, for unnotched specimens, between results at $R = +0.25$ (No. 14, Table IV) and results at $R = +0.02$ (not shown in Table IV—see reference cited).

EFFECTS OF NOTCHES IN PLATE BENDING AND IN REPEATED TORSION TESTS

Tables II and III, showing information from plate-bending fatigue tests and from torsion fatigue tests, are included mainly to illustrate the meager extent of such data in comparison to those from tests under other types of loading. Results available for inclusion in these two tables are too few to warrant discussion, although it may be observed that values of K_f are again generally less than values of K_T.

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[3] Until the size effect in fatigue is better understood, unnotched fatigue strength values should be chosen for the size of notched member under consideration.

TABLE VI.—SOME FATIGUE-STRENGTH REDUCTION FACTORS FROM AXIAL-LOADING FATIGUE TESTS AT NON-ZERO MEAN STRESSES. (FROM DATA IN TABLE IV.)

	K_T	Values at 10^5 Cycles				Values at 10^7 Cycles			
		Mean Stress, psi.	$K_f{}^b$			Mean Stress, psi.	$K_f{}^b$		
			K_f'	K_f''	K_f'''		K_f'	K_f''	K_f'''
				24 S-T					
No. 9	2.6	37 500	2.56	1.53	1.31	32 000	2.50	1.73	1.30
No. 10	2.6	33 000	...	1.55	...	27 000	2.00	1 83	1.25
No 11	2.6	27 300	...	1.72	...	24 500	1.84	1.77	1.26
No. 12	2.6	20 600	...	1.82	...	15 100	...	2.08	...
				24 S-T ALCLAD					
No. 13	2.6	48 100	1.94	1.22	1.12	23 800	2.44	1.20	..
No 14	2.6	33 600	2.10	1.50	1.22	17 600	1.84	1.22	...
No. 15	2.6	25 200	1.77	1.56	1.23	12 200^a	1.66^a	1.26^a	...
No. 16	2.6	18 200	1.33	...	...	8 100^a	...	...	...
No. 17	2.6	12 800	1.50	1.60	...	5 900^a	1.83^a	...	...
No 18	2.6	8 000	1.65	1.77	...	4 000^a	1.71^a	...	...
No. 19	2.6	3 900	...	2.32^a	...	2 200^a	1.78^a	...	...

[a] Denotes case where maximum stress was less than yield strength.
[b] See Table V for definitions of K_f', K_f'', and K_f'''.

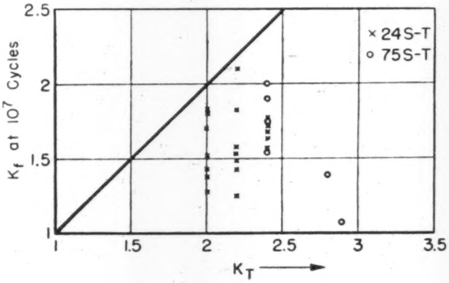

FIG. 3.—Variation of Fatigue-Strength Reduction Factor, K_f at 10^7 Cycles with K_T for Reversed Axial-Loading Tests of Sheet Specimens with Center Holes. (Table IV Items 1 to 8 and 24 to 27.)

EFFECTS OF NOTCHES IN REPEATED AXIAL LOADING

Table IV lists values obtained from axial loading fatigue tests. It may be noted that (with one exception) only aluminum alloys are listed and that all values reported are from tests on sheet specimens; relatively few axial-loading notch-fatigue data appear to be available for structural materials in general.

Item Nos. 1 through 9 and 21 through 24 are results of axial loading fatigue tests in which the nominal stress was fully reversed each cycle; for these, a fatigue strength reduction factor, K_f, may be defined in the same manner as for rotating-beam fatigue tests. Figure 3 shows a plot of K_f (at 10^7 cycles) against K_T for the results of fully-reversed axial-loading tests; this shows two features in

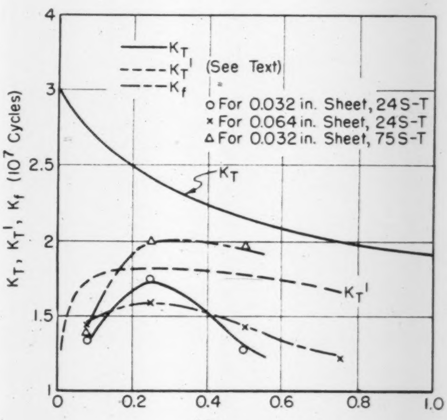

FIG. 4.—Variation of K_f at 10^7 Cycles for Reversed Axial-Loading Fatigue Tests of Sheets with Center Holes, with Ratio of Hole Diameter to Sheet Width for $\frac{1}{4}$-in. Wide 24S-T Sheet. (See Table IV.)

common with Fig. 1 for results of rotating-beam tests:

1. K_f appears less than K_T, and
2. K_f/K_T decreases as K_T increases.

Somewhat similar observations are possible for K_f at 10^5 cycles, although for these data, one may not conclude that K_f at 10^5 cycles is always less than K_f at 10^7 cycles. As Brueggeman and Mayer (9) have pointed out, these test results show another interesting feature if K_f is plotted against the ratio d/w (hole diameter to sheet width) for fixed sheet width. This is illustrated in Fig. 4.[4] It is particularly interesting that, for very small-diameter holes, K_f is small. Thus, the fatigue strength reduction decreases toward unity as the size of the notch decreases toward zero.

Analyses of notch-fatigue tests in which the nominal stress cycles are not fully reversed requires careful attention to the definition of the term "fatigue strength reduction factor." The illustrative example in Table V shows three possible definitions of K_f for loading conditions such that the nominal mean stress differs from zero. Clearly, other definitions are possible; the three shown include those that have been rather generally advocated in the literature. Table VI lists values of each of these differently defined fatigue-strength reduction factors, for two lifetimes, for the test results in Table IV. The following points are of interest: (1) For any of these definitions, K_f is less than K_T for all entries and (2) while available data are too few for adequate discrimination, K_f''' is generally smallest of the three. This second observation suggests that most conservative design might be effected by applying the theoretical stress-concentration factor to the maximum stress for an unnotched specimen having the same lifetime *at the same nominal mean stress* as the notched specimen under consideration. (For the data tabulated in Table VI, K'_f is generally closest to K_T; some unpublished results indicate that, in certain instances, use of this may be slightly unconservative.)

THE FATIGUE NOTCH SENSITIVITIES OF MATERIALS

The illustrative experimental data reviewed in previous sections indicate that geometrical notches usually reduce the fatigue strength of a member. In general, the fatigue strength reduction factor, K_f, increases with the severity of the notch (as measured by the stress-concentration factor, K_T); however, beyond the observation that $K_f \leq K_T$, plots (such as Figs. 1 through 3) of results assembled from a number of sources do not show any simple relation valid for different materials.

It is commonly believed that one reason K_f is often less than K_T is that local yielding and work hardening decrease the peak stresses under a notch below the value predicted by elastic theory and strengthens this region of the member. It then seems plausible to expect that different materials, having different characteristics, would show different relations between K_f and K_T. It has been suggested that such behavior should be expressible by some sort of notch-sensitivity index. One of the simplest of suggested indices is

$$q \equiv \frac{K_f - 1}{K_T - 1}$$

According to this, a material which shows no reduction in fatigue strength due to a notch ($K_f = 1$) would have a notch-sensitivity index $q = 0$; a material which shows as much reduction as predicted

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[4] This figure includes a plot of values of K'_T, the "Technical stress-concentration factor" advocated by Neuber. In this instance, the value 0.0189 in. suggested by Neuber, was used for the material constant concerned. While it is interesting to note that this K'_T predicts low values at small d/w, it should be mentioned that Neuber's hypothesis has not been wholly successful in other cases.

(on a maximum-stress theory of failure) by the theoretical stress-concentration factor ($K_f = K_T$) would have a notch-sensitivity index $q = 1$.

Table VII gives values of q (for a lifetime of 10^7 cycles) for all rotating-beam fatigue test results listed in Table I. Examination indicates:

TABLE VII.—VALUES OF K_f AND q FROM ROTATING-BEAM DATA IN TABLE I.

	Material	Tensile Strength, psi.	K_T	K_f at 10^7 Cycles	q_f at 10^7 Cycles
No. 1....	SAE 4130	76 200	2.6	1.7	0.44
No. 2....	SAE 4130	139 500	2.6	2.9	1.17
No. 3....	SAE 4130	199 300	2.6	2.9	0.72
No. 4....	SAE 4130	141 800	2.0	1.9	0.95
No. 5....	SAE 4130	141 800	2.0	1.9	0.87
No. 6....	SAE 4130	141 800	2.0	1.8	0.83
No. 7....	SAE 4130	141 800	2.0	1.8	0.84
No. 8....	SAE 4130	141 800	2.0	1.8	0.82
No. 9....	SAE 4130	104 300	2.9	2.0	0.55
No. 10....	SAE 4130	151 500	2.9	2.7	0.91
No 11....	SAE 4130	185 800	2.9	2.3	0.67
No. 12....	SAE 4140	133 000	3.1	2.6	0.78
	SAE 4140	133 000	2.1	2.1	0.51
	SAE 4140	133 000	1.6	1.6	1.00
No. 13....	SAE 4140	275 500	3.1	2.0	0.45
	SAE 4140	275 500	2.1	1.6	0.51
	SAE 4140	275 500	1.6	1.2	0.28
No. 14....	SAE 4340	163 700	2.0	1.6	0.59
No. 15....	SAE 4340	163 700	2.0	1.7	0.69
No. 16....	SAE 4340	163 700	2.0	1.6	0.62
No. 17....	SAE 4340	163 700	2.0	1.6	0.61
No. 18....	SAE 4340	163 700	2.0	1.8	0.76
No. 19....	24 S-T	83 000	2.6	2.4	0.85
No. 20....	24 S-T	68 000	2.0	1.9	0.85
	24 S-T	68 000	5.0	2.3	0.32
No. 21....	75 S-T	86 200	2.0	2.3	1.31
No. 22....	75 S-T	86 200	2.0	1.9	0.88
No. 23....	75 S-T	86 200	2.0	1.8	0.79
No. 24....	75 S-T	86 200	2.0	1.8	0.79
No. 25....	75 S-T	86 200	2.0	1.5	0.51
No. 26....	18-8 annealed	82 800	2.6	0.81	−0.12
No 27....	18-8	132 500	2.6	1.43	0.27

1. Values of q vary nearly as much among results of different tests on one material (note Item Nos. 1 through 6 for S.A.E. 4130 and Item Nos. 26 and 27 for 18-8) as among different materials and
2. While trends are evident within limited groups of data (note apparent effect of hardness for Item Nos. 12 and 13), such trends do not appear to hold for all data listed.

Investigation of these and other data has, to date, shown no systematic variation of the index q with hardness, or yield point, or yield ratio that holds for a wide range of results assembled from many sources. It appears that q (or any simple relation involving only K_f and K_T) may be inadequate as a general fatigue notch-sensitivity index for structural materials.

Consideration of factors which may be expected to influence the response of a material to notches suggests a number of reasons why available data can hardly be expected to afford valid fatigue notch-sensitivity indices for materials and indicates some types of investigation which might lead toward more useful information than now exists.

Probably the most important factor resulting from the presence of a notch is the existence of *localized peak stresses* exceeding the nominal stress. Magnitudes of peak stresses are commonly estimated in terms of the nominal stress imposed and the approximate stress-concentration factor. In comparing results from different sources, however, there are often additional factors difficult to evaluate. For example:

1. Values of K_T are not known with equal precision for different notches. For most actual cases, available theory is approximate; use of different types of notches may involve different types of approximation in evaluation of K_T. In some instances, the precision of notch form actually machined may be questionable; not all reported data give information adequate to judge this. In general, some recent papers include careful consideration of estimation of K_T but many early reports leave this point in question.
2. Machining and polishing a sharp notch may leave the surface at the root of the notch in a different state

(for example, with more cold work) than that of the comparison unnotched specimen.[5]

3. Peak local stresses due to notches are not generally expected to exceed the yield point of the material under consideration; values estimated from stress-concentration factors deserve special attention when they are high compared to material yield stresses. Examination of Tables I through IV suggests that such high peaks are uncommon in rotating-bending tests except for specimens of low yield-strength material or specimens with very severe notches; that high peak stresses are relatively common in axial-loading tests at high nominal mean stress.

For such reasons as these, comparison of results obtained with different notch forms and different surface finishes do not afford basic knowledge of material properties.

There appears to be cumulative evidence that not only peak stresses but also *local stress distribution* may affect the fatigue strength of a notched member. Figure 4 illustrates the low ratio of K_f to K_T for sheet specimens notched by small holes. Item No. 20 in Table I shows a similar small ratio for a sharp V-groove in a rotating-beam specimen. Other scattered results also indicate that very small notches may be less serious than implied by their stress-concentration factors. A number of ideas have been advanced to explain such "size effects of notches": that not peak stress but average stress over some small region is critical; that failure may start below the surface; that stress gradient is a factor; that there is a statistical size effect; that fatigue failure depends on stresses transverse to the peak stress, etc. The point to be emphasized here is that available experimental data suitable to examine the importance of local stress-distribution effects are meager. With the exception of results for aluminum alloy sheets with holes, there seem to be few reported investigations in which either the severity of a particular type of notch or the nature of notches of a particular severity (K_T) has been systematically varied over a very wide range for any one material and kind of loading.

Notch-fatigue test data now available for most structural materials appear inadequate, both in nature and extent, to separate effects characteristics of material properties from effects that may be due to variation among the many notch forms, notch severities, surface preparations, and loading conditions that have been used. As Paterson (12) suggests, there is need for systematic investigation on such aspects as: the influence of K_T on K_f for significant ranges of K_T, the influence of relative sizes of notches of similar K_T, detailed stress analyses of notch forms, and criteria of multi-axial stress conditions. Until more basic information of this kind is available, it seems unwise to characterize materials by any sort of fatigue notch-sensitivity indices with the expectation that such indices will be generally valid in design.

CONCLUSIONS

1. Examination of data assembled from various sources has not afforded characterization of fatigue notch sensitivities of a number of materials of interest in aircraft structures.

2. Available information seems inadequate, not only in extent but also in

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[5] Attention to this is increasing, and different expedients have been considered for comparison of unnotched and notched specimens. H. A. Wills mentioned (in private discussion) experiments at Melbourne in which deep notches are cut by a sharp tool and not thereafter polished but fatigue strengths of notched specimens are compared to fatigue strengths of unnotched specimens with a machine finish. In some work in progress at Battelle, electropolishing is being used as a means of obtaining comparable surface finish on unnotched and on notched fatigue test specimens.

nature of notch fatigue test investigations of materials.

3. While the assembled data, therefore, do not afford rules for accurate design, they suggest that the following principles may be conservative:

(*a*) For completely reversed stress loading, apply appropriate theoretical stress-concentration factors to fatigue strengths of unnotched specimens.

(*b*) For partly reversed stress loading, apply appropriate theoretical stress-concentration factors to maximum stress values for unnotched specimens having the same lifetime at the same nominal mean stress.

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(1) T. T. Oberg and J. B. Johnson, "Fatigue Properties of Metals used in Aircraft Construction at 4350 and 10600 Cycles," *Proceedings*, Am. Soc. Testing Mats., Vol. 37, pp. 195–205 (1937).

(2) H. F. Moore and D. Morkovin, "Progress Report on the Effect of Size of Specimen on Fatigue Strengths of Three Types of Steel," *Proceedings*, Am. Soc. Testing Mats., Vol. 42, pp. 145–154 (1942). Also, Second Progress Report on same, *Proceedings*, Am. Soc. Testing Mats., Vol. 43, pp. 109–124 (1943).

(3) H. J. Grover, R. W. Bennett, and G. M. Foley, "Fatigue Properties of Flash Welds," *Welding Journal, Research Supplement*, November, 1945, pp. 599S–618S.

(4) Unpublished data, Battelle Memorial Institute.

(5) T. J. Dolan and B. C. Hanley, "Effect of Size and Notch Sensitivity on Fatigue Characteristics of Two Metallic Materials," Part II, *AF Technical Report, 5726*, October, 1948, pp. 78–153.

(6) G. H. Found, "The Notch Sensitivity in Fatigue Loading of Some Magnesium Base and Aluminum Base Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, pp. 715–740 (1946).

(7) H. F. Moore, "Effect of Size and Notch Sensitivity on Fatigue Characteristics of Two Metallic Materials," Part I, *AF Technical Report 5726*, October, 1948, pp. 1–77.

(8) W. C. Brueggeman, M. Mayer, Jr., and W. H. Smith, "Axial Fatigue Tests at Zero Mean Stress of 24S-T Aluminum Alloy Sheet With and Without a Circular Hole," *Technical Note No. 955*, Nat. Adv. Committee Aeronautics, November, 1944.

(9) W. C. Brueggeman and M. Mayer, Jr. "Axial Fatigue Tests at Zero Mean Stress of 24S-T and 75S-T Aluminum Alloy Strips With a Central Circular Hole," *Technical Note No. 1611*, Nat. Adv. Committee Aeronautics, August, 1948.

(10) H. W. Russell, L. R. Jackson, H. J. Grover, and W. W. Beaver, "Fatigue Strength and Related Characteristics of Aircraft Joints," *Technical Note No. 1485*, Nat. Adv. Committee Aeronautics, February, 1948.

(11) H. Neuber, "Theory of Notch Stresses," *Translation No. 74*, David Taylor Model Basin, November, 1945.

(12) M. S. Paterson, "Notch Sensitivity of Metals, the Failure of Metals by Fatigue," Melbourne University Press (1947).

DISCUSSION

Mr. R. E. Peterson[1].—Mr. Grover points out that the q values vary greatly for a given material. While it might be hoped that this would turn out to be constant, there are reasons why this should not be expected. For a given material (given grain size and structure) one would expect from probability considerations[2] that failure should be a function of the volume of material near peak stress, S_{max} (Fig. 5). This can be expressed in terms of stress gradient[3], which is roughly proportional to the notch radius, r. Plots of q *versus* r show[4] (Fig. 6) small q values for small radii and larger q values, in some cases approaching 1.0, for larger radii.

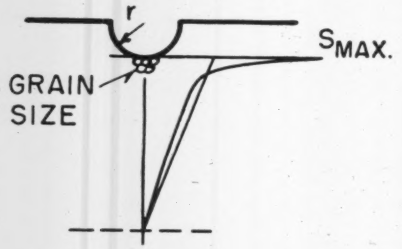

Fig. 5.

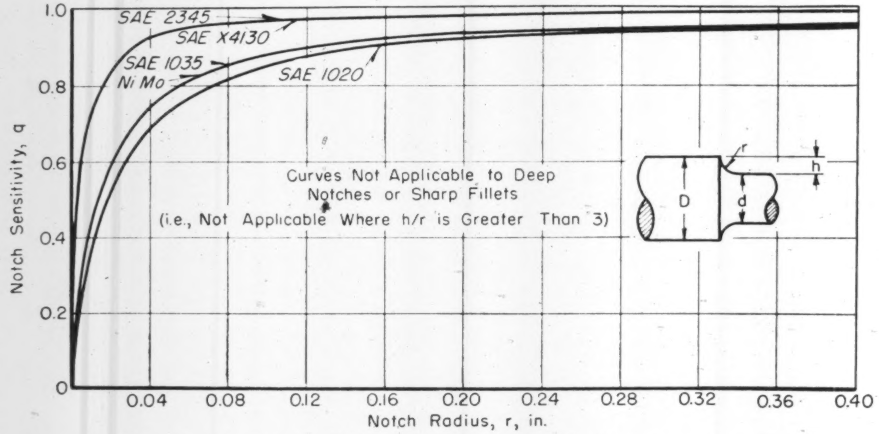

Fig. 6.—Ratio of Notch Sensitivity, q, to Notch Radius, r.

The data given in Mr. Grover's paper on 75S-T and 24S-T aluminum alloy sheets containing a central hole[5] seem to show the above-mentioned trend (Fig. 7). In computing q values, the basic endurance strength value for the unnotched specimen should, from probability considerations[2], correspond to the largest specimen available; 17,000 psi. was used for 75S-T and 18,000 psi. for 24S-T in all calculations. Referring to

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[1] Manager Mechanics Division, Westinghouse Research Laboratories, East Pittsburgh, Pa.

[2] Properties of Metals in Materials Engineering, Am. Soc. Metals, Cleveland, Ohio, p. 68 (1949).

[3] Stephen Timoshenko 60th Anniversary Volume, Macmillan Co., p. 180 (1938).

[4] R. E. Peterson, "Relation between Life Testing and Conventional Tests of Materials," ASTM Bulletin, No. 133, March, 1945, p. 13.

[5] W.C. Brueggeman and M. Mayer, "Axial Fatigue Test at Zero Mean Stress of 24S-T and 75S-T Aluminum Alloy Strips with a Central Circular Hole," *Technical Note 1611*, Nat. Adv. Committee Aeronautics.

Fig. 7, one would consider the average curve in comparing behavior of various materials; for design purposes the upper curve would be the "safe" one to use.

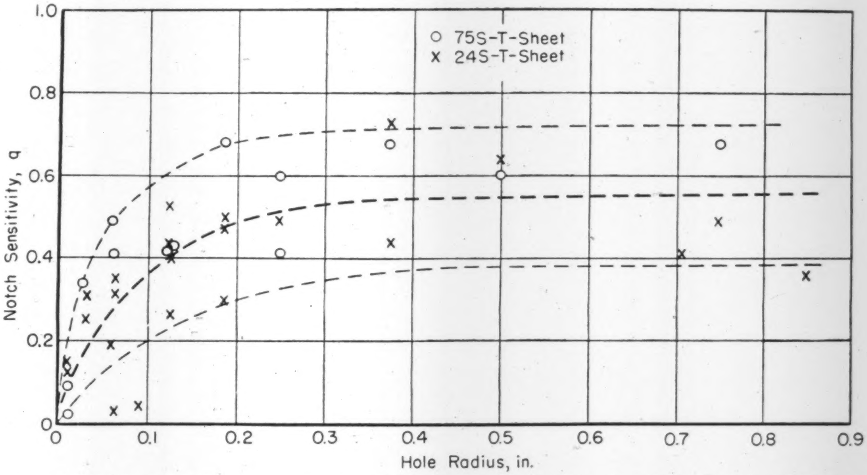

FIG. 7.—"q" Values for Flat Strips Having a Transverse Hole. (Based on Nat. Bureau of Standards Data-Alumium Alloys.)

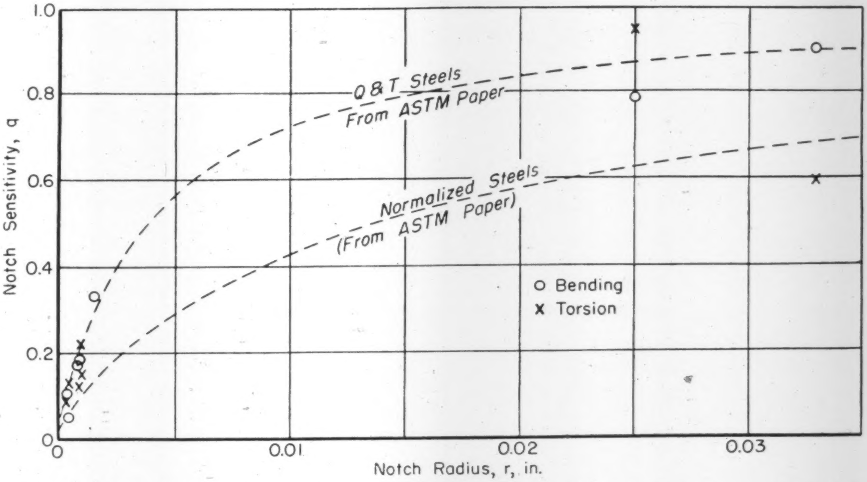

FIG. 8.—"q" Values for Test Bars with Grooves, Holes, Shoulder Fillets. (Based on British Data—Heat-Treated Alloy Steels.)

Strictly speaking, a more logical abscissa variable would be the volume of material at peak stress[3] (assuming probability to be the governing consideration), but there is so much work in determining this that the method would not be useful for ordinary design purposes. This is why the "notch" radius, r, which is known, is used. This is also why the curves are labeled 24S-T and 75S-T sheet, rather than merely 24S-T and 75S-T, independent of form.

Recently extensive British work[6] on combined stress has become available. This is particularly valuable in that it includes tests of notches of very small radius (less than 0.001 in.). It was interesting to find (Fig. 8) that these show the same general tendency described in the foregoing.

Mr. Grover's paper presents a large amount of useful material in a clear and orderly manner, and this will be of great value to the airplane designer.

MESSRS. D. ROSENTHAL[7] and G. SINES.[7]—It is indeed heartening to see that such an intensive research on the subject of notch fatigue is in progress. This large quantity of data taken under identical conditions on carefully identified notches should make closer examination of the theories feasible.

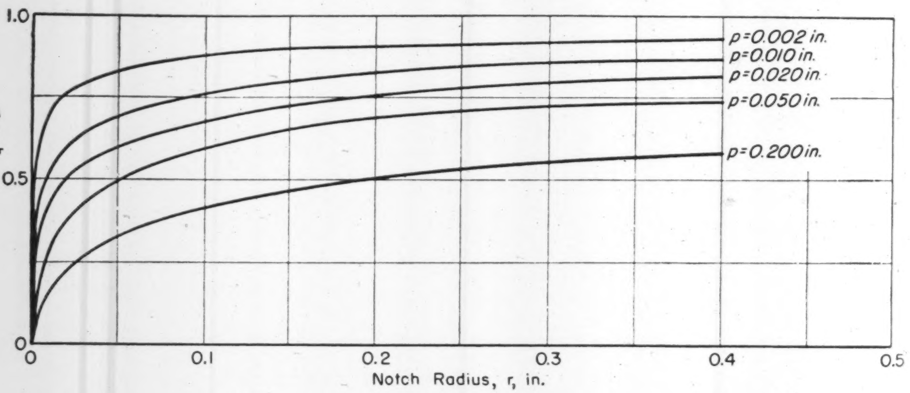

FIG. 9.—Variation of q' (Based on Neuber's Technical Stress-Concentration Factor) with Notch Radius, r.

One aspect of this problem, the effect that residual stress at the notch has on the fatigue life, is being studied at the University of California at Los Angeles. As Mr. Grover has stated, stress discontinuities in machine parts and at the welds have been the points of initiation of many fatigue cracks that have continued to advance and cause complete failure of the structure. In machine parts that have been heat treated or welded there are usually residual stresses, often of high magnitude, induced at the notches. The loading cycle itself plays a large rôle in the residual stress picture. It may relieve or increase the residual stresses present in a part or impart residual stresses to an initially stress-free part. These residual stresses may be either beneficial or deleterious. Residual stresses can also be induced intentionally by many means to increase the life of a machine component subjected to repeated stress.

We should like to present briefly a few of the test results that we have already obtained in this study:

The tests were performed on an aluminum alloy 61 S, in a fully hardened condition, 61 S-T, and also in a partially softened condition. For each of these conditions three sets of specimens were tested: (*a*) without notch, (*b*) notched, but with no residual compression, and (*c*) notched, with residual compression. The residual compressive stress at the notch was set up by deforming plastically the region around the notch in tension. The amount of deformation was deemed to be sufficiently small to neglect the effect of strain hardening.

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[6] H. J. Gough, "Engineering Steels under Combined Cyclic and Static Stresses," Presidential Address, Institution of Mechanical Engrs., October 21, 1949 (London).

[7] University of California, Los Angeles, Calif.

Two series of fatigue tests were performed, one with complete reversal of stress and the other under relieved loading, that is, the variation of stress from zero to a tensile stress.

For the particular type of notch, no benefit from the residual compression induced by prestraining was found in the partially softened aluminum alloy for which the ratio of yield to fatigue stress was rather low (1.5). X-ray diffraction stress measurements showed that in consequence of this low ratio all residual compression was practically removed in reverse bending, while in relieved bending an amount of residual stress nearly equal to that induced by prestraining was set up in the stress-free specimens by the loading itself. On the other hand, in the fully-hardened aluminum alloy with a high yield-fatigue stress ratio (3.0) an improvement up to 30 per cent of the notch-fatigue stress was obtained in both reversed and relieved bending due to residual compression for the same notch. This result agrees with the X-ray stress measurements which showed no change of residual compression in reversed bending even after 10^7 cycles at the stress slightly below the endurance limit.

The above findings also were in reasonable agreement with values computed from a modified Goodman diagram for the material and the strength reduction ratio for the type of notch investigated.

Tests are now in progress to extend the investigation by studying the effect of residual tensile stresses.

MR. H. J. GROVER (*author's closure*).—Mr. Peterson points out that q values vary with notch radius, r, and he presents some interesting examples of the tend of this variation in specific cases. He suggests that this variation of q with r may be due to variation of the volume of material near peak stress.

It is interesting to note an alternative explanation of some, at least, of the variation of K_f from K_T Neuber suggests that anisotropy of metals in regions of sharp stress gradients would lead to an effective stress-concentration factor

$$K_N = 1 + \frac{K_T - 1}{1 + \sqrt{\rho/r}}$$

where ρ is a value characteristic of the metal under test. If one supposes that the fatigue strength reduction factor, K_f, may be indentified with K_N, then one may define a quantity q' as follows:

$$q' = \frac{K_N - 1}{K_T - 1} = \frac{1}{1 + \sqrt{\rho/r}}$$

Figure 9 shows a plot of q' *versus* r for several values of ρ. General similarity of the shapes of these curves with those of curves in Mr. Peterson's Figs. 6, 7, and 8 is apparent.

It is entirely possible that neither probability considerations connected with the volume of material at peak stress nor Neuber's concept of effects of anisotropy in regions of stress gradients contain all of the factors important in determining the fatigue-strength reduction of a notched part. In any event, it seems important that, until this situation is more completely understood, the design engineer avoid using one value of any present index of fatigue-notch sensitivity as a qualification of a material applicable to all cases of design against fatigue failure. As Mr. Peterson shows, a value of q for a notch of small radius may be unsafe for design of a part with a notch of the same type, but of larger radius. Moreover, present information seems inadequate to tell to what extent q may be constant for one material, in one condition, with notches of the same root radius but of different character (for example, fillets compared to grooves or to transverse holes).

Messrs. Rosenthal and Sines have emphasized another factor of considerable importance in fatigue-strength reduction–residual stress at the root of a notch. As they note, such stress may be induced either in fabrication or preloading of a specimen (or structural part), or in the loading cycle of the fatigue test (or service loading) itself. This is another reason why an index such as the q value is not simple to use in design for all kinds of cyclic loading. One may expect plastic flow at the root of a notch under a load such that the material there exceeds its yield stress. Such local deformation may: (1) produce an effective stress-concentration factor, at the peak of the loading cycle, less than K_T, and (2) leave residual stress on unloading. Under such conditions, as one might expect, one often finds K_f less than under cyclic loading at lower nominal maximum stress. Examination of Tables I to IV in the paper indicates that such local yielding may occur: (1) under fully reversed loading, mainly in the finite-lifetime region but (2) under tension-tension loading, at high R, even at long lifetimes.

The author would like to express his appreciation to Mr. Peterson and to Messrs. Rosenthal and Sines for their helpful comments and discussion.

EFFECTS OF ANODIC COATINGS ON THE FATIGUE STRENGTH OF ALUMINUM ALLOYS[*][1]

By G. W. Stickley[2] and F. M. Howell[2]

Anodic treatments of aluminum alloys produce coatings of aluminum oxide that have various desirable properties. The coatings are hard, providing improved resistance to abrasion. They are adherent, providing good protection against the weather and chemical attack; and where such protection is the main consideration, those coatings may be impregnated with a corrosion inhibitor and also serve as an excellent base for paint.

There are anodic treatments in considerable variety, each designed to provide certain useful combinations of properties. The properties of the oxide coatings will vary not only with the treatment but also with the composition and structure of the alloy being treated. Some of the variations in characteristics result from the differences in thickness of coating. Others result from the fact that during anodic treatment some constituents in the alloy are dissolved or oxidized while other constituents are not.[3, 4]

The effects of anodic treatments upon the tensile properties of the coated articles generally are so small as to be well within the range of variation in testing. In structural parts subjected to repeated loads, however, it is possible that the effects on fatigue resistance may sometimes be significant.

The effects of anodic coatings on the fatigue strengths of aluminum alloys have been studied by several investigators. Gerard and Sutton[5] reported that at 50 million cycles the fatigue strength of duralumin was increased from 20,200 psi. to 24,600 psi. when an anodic coating was applied,[6] while Mueller[5] found that an anodic coating reduced the fatigue strength (cycles not stated) of Avional about 20 per cent.[7] In another case, Mueller reported an anodic coating had no effect on the fatigue strength.[8] Mauksch reported a slight improvement at large numbers of cycles for an aluminum-magnesium-silicon alloy with an anodic coating 0.0004 in. thick. He stated further that "if, for instance, the tests had been carried out for only 3 million stress reversals, it could have been stated that the endurance strength of the anodically-coated bars was considerably lower." In some other tests, Mauksch found that the fatigue strength of duralumin ZB1/3 at 10 to 12 million cycles was increased from 22,000 psi. to 23,000 psi. by coatings 0.00015 to 0.00045 in. in thickness. Further tests of duralumin 681B by Mauksch with coatings 0.00025

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[*] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] This paper which is a contribution to the work of Subcommittee VI on Anodic Oxidation of Aluminum and Aluminum Alloys of Committee B-7 on Light Metals and Alloys, Cast and Wrought was prepared at the request of Committee E-9 on Fatigue.

[2] Assistant Chief and Chief, respectively, Mechanical Testing Division, Aluminum Research Laboratories, New Kensington, Pa.

[3] F. Keller and G. W. Wilcox, "Anodically Oxidized Aluminum Alloys—Metallographic Examination," *Metals and Alloys*, Vol. 10, June, 1939., p. 187.

[4] F. Keller, G. W. Wilcox, M. Tosterud, and C. J. Slunder, "Anodic Coating of Aluminum—Behavior of Alloy Constituents," *Metals and Alloys*, Vol. 10, July, 1939, p. 219.

[5] "Bending Fatigue Strength of Anodized Aluminum," *Light Metals* (London), Vol. V, June, 1942, p. 223.

[6] Although the thickness of the coating is not stated, the details of the treatment indicate that a coating about 0.0001 in. thick would have been produced.

[7] Thickness of coating not stated, but since it was produced in a sulfuric acid solution it was probably relatively thick.

[8] Thickness of coating not stated, but since it was produced in a chromic acid solution it was probably relatively thin.

TABLE I.—COMPOSITION OF ALLOYS.

Alloy	Composition, per cent					
	Copper	Silicon	Manganese	Magnesium	Zinc	Chromium
17S	4.0	...	0.5	0.5	...	...
25S	4.5	0.8	0.8	...	...	...
75S	1.6	...	...	2.5	5.6	0.3
B355	1.3	5.0	...	0.5	...	0.15

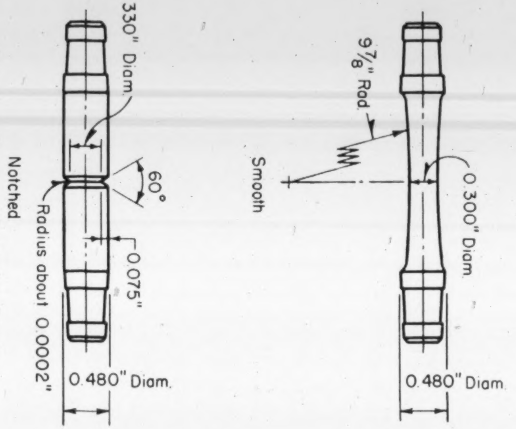

FIG. 1.—Types of Rotating-Beam Fatigue Specimens.

in. thick also indicated slight improvement. Dolan[9] found that anodizing (thickness of coating not stated) had no effect on the fatigue strength of 25S-T6, but raised the endurance limit (500 million cycles) of 76S-T6 about 3000 psi. (14 per cent). Henshaw, Wallerstein and Zand[10] reported the results of tests of some 75S-T6 aircraft-engine-mounting components with anodic finishes; these data indicated that coatings 0.0004 to 0.0008 in. thick reduced the fatigue strength, but coatings 0.00004 to 0.00008 in. thick apparently did not.

In the study of certain applications in-

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[9] Thomas J. Dolan, "Certain Mechanical Strength Properties of Aluminum Alloys 25S-T and X76S-T," N.A.C.A. *Technical Note No. 914*, Nat. Adv. Committee for Aeronautics, October, 1943.

[10] R. C. Henshaw, Leon Wallerstein, Jr., and S. J. Zand, "Fatigue Life of Aircraft Engine Mounting Components," presented at the S.A.E. Annual Meeting, Detroit, Mich., January, 1950.

TABLE II.—MATERIALS AND COATINGS TESTED.

Alloy and Temper	Product	Size, in.	Type of Test	Specimen		Anodic Treatment					
				Direction	Surface	Alumilite[a] No.	Thickness of Coating, in.	Electrolyte	Current, amp. per sq. ft.	Time, min.	Seal
17S-T3	Sheet	0.064	Repeated flexure	Transverse	As rolled	205	0.0002	15 per cent sulfuric acid	12	15	5 per cent dichromate solution, 15 min.
17S-T4	Rolled-and-drawn rod	¾	Rotating beam	Longitudinal	Smooth, polished						
25S-T6	Forged slab	¾ by 6	Rotating beam	Longitudinal and transverse	Smooth, polished	Black 100	0.0010	15 per cent sulfuric acid	16	..	Pigmented black
75S-T6	Rolled-and-drawn rod	¾	Rotating beam	Longitudinal	Smooth, polished Sharp V-notch	1224	0.00009	3 per cent chromic acid	1 to 3	30 to 60	None
					Smooth, polished Sharp V-notch	204	0.0005	15 per cent sulfuric acid	12	30	Water immersion at 210 F., 15 min.
75S-T6	Extrusion	¾ by 2	Rotating beam	Longitudinal	Smooth, polished	1224	0.00009	3 per cent chromic acid	1 to 3	30 to 60	None
					Smooth, polished	204	0.0005	15 per cent sulfuric acid	12	30	Water immersion at 210 F., 15 min.
B355-T6	Sand-cast test bars	½	Rotating beam	...	Smooth, polished	704	0.00015	15 per cent sulfuric acid	9	30	Water immersion at 210 F., 15 min.

[a] The Alumilite treatments are patented.

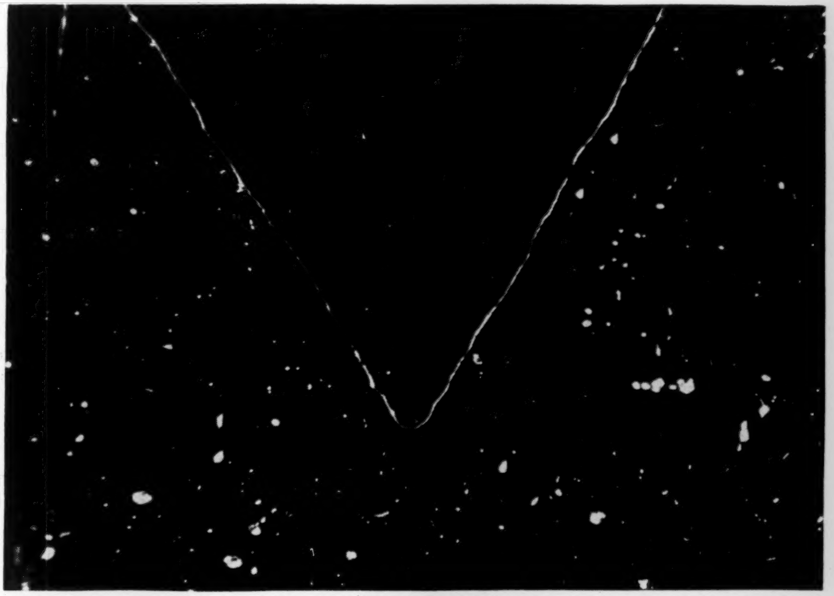

FIG. 2.—Cross-Section of 75S-T6 Alloy Notched Fatigue Specimen, As Machined. Dark Field Illumination ×500.

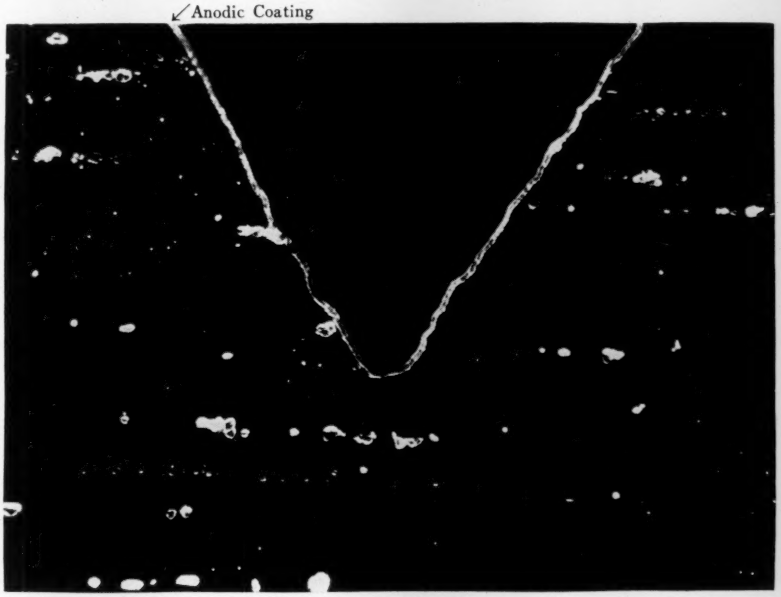

FIG. 3.—Cross-Section of 75S-T6 Alloy Notched Fatigue Specimen After Alumilite 1224 Treatment. Dark Field Illumination ×500.

volving anodic coatings, Aluminum Research Laboratories has at various times made fatigue tests involving the use of a particular anodic treatment for a given alloy. The results of these tests are presented in this paper. They, of course, represent only a few of the many combinations of alloys and treatments that might be used. Also, they are not necessarily an indication of the effects that will obtain when using other conditions of anodic treatment or when using a different aluminum alloy.

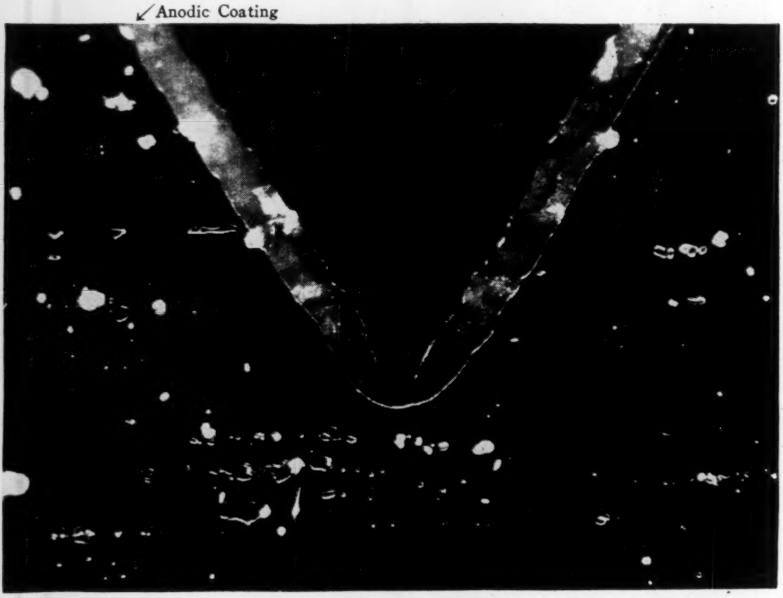

FIG. 4.—Cross-Section of 75S-T6 Alloy Notched Fatigue Specimen After Alumilite 204 Treatment. Dark Field Illumination ×500.

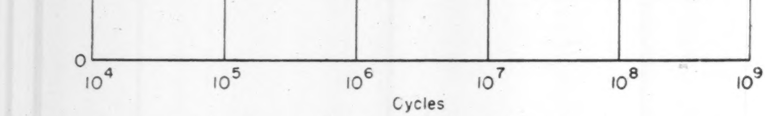

FIG. 5.—Flexural Fatigue Data for 17S-T3 Sheet.

MATERIAL

The tests involved a total of four alloys (17S, 25S, 75S and B355), three in wrought forms and one in the form of sand-cast test bars. Five different anodic

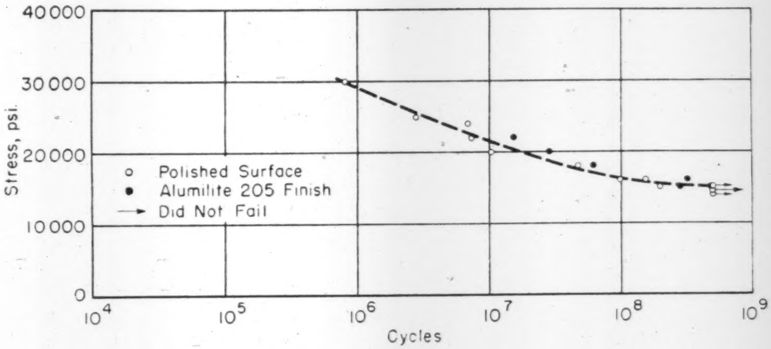

FIG. 6.—Rotating-Beam Fatigue Data for 17S-T4 Rolled-and-Drawn Rod.

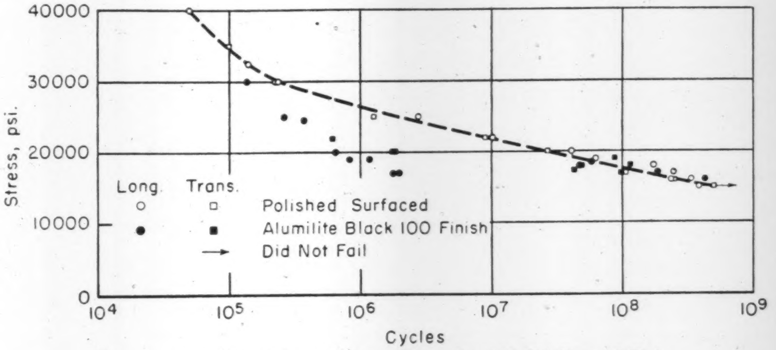

FIG. 7.—Rotating-Beam Fatigue Data for 25S-T6 Forged Slab.

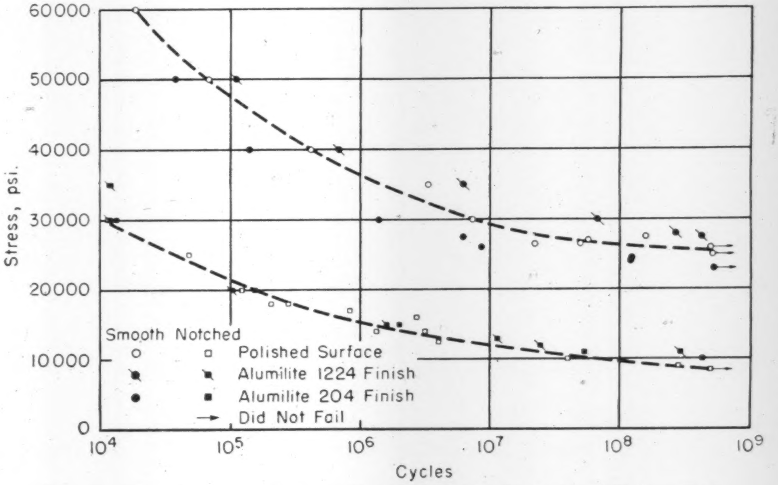

FIG. 8.—Rotating-Beam Fatigue Data for 75S-T6 Rolled-and-Drawn Rod.

treatments were employed, but only one alloy (75S) was tested with more than one type of anodic treatment. The thicknesses of the oxide coatings ranged from about 0.0001 to 0.0010 in. The compositions of the several alloys tested are shown in Table I, and the products[11] and the anodic treatments used are shown in Table II.

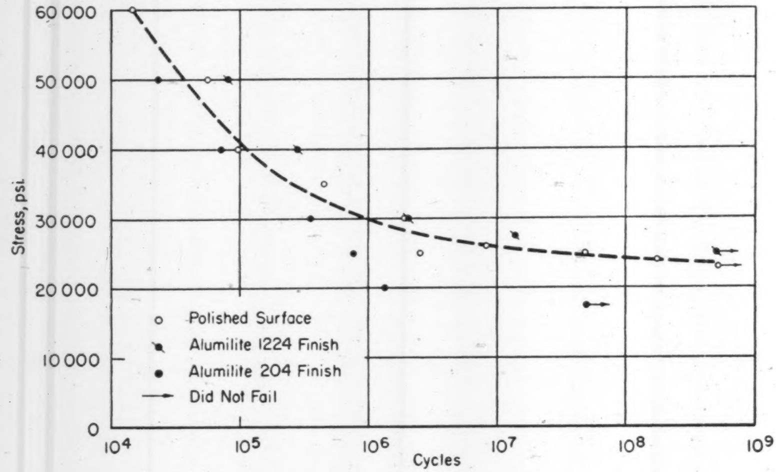

FIG. 9.—Rotating-Beam Fatigue Data for 75S-T6 Extruded Bar.

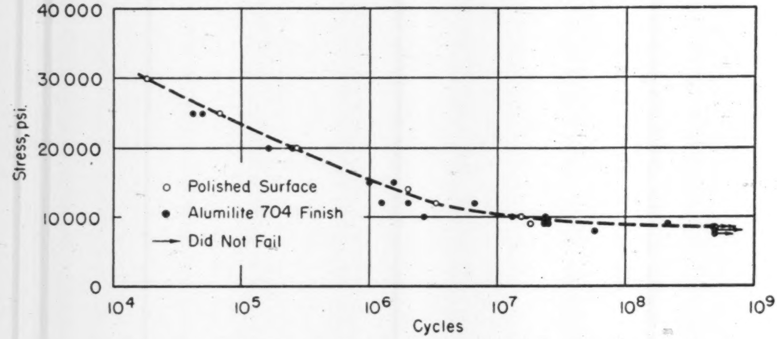

FIG. 10.—Rotating-Beam Fatigue Data for B355-T6 Sand-Cast Test Bars.

EQUIPMENT AND METHODS OF TEST

Tests of the 17S-T3 alloy sheet were made with the Aluminum Research Laboratories' constant-deflection repeated-flexure machine, using a cantilever specimen subjected to reversed bending stresses at 1500 cpm., as previously described.[12] Tests of the other products were made in R. R. Moore rotating simple-beam machines at speeds of 3500 and 10,000 cpm., using smooth specimens. In addition, rotating-beam tests of the 75S-T6 rod were made, using specimens containing a sharp V-notch. The two designs of rotating-beam specimens are shown in Fig. 1.

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[11] The fatigue properties of the 17S and 25S alloys are not representative of these alloys as currently produced. Improvements in fabrication practices since these particular lots were made have resulted in consistently higher values for the endurance limits of these alloys. The evaluation of the coating is, however, unaffected by this fact.

[12] R. L. Templin, "The Fatigue Properties of Light Metals and Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, pp. 364–380 (1933).

Sheet specimens for these tests were machined in packs to the required contour by cross milling, using a profile cutter. To minimize the possibility of any notches at the edges, the corners were rounded slightly by polishing longitudinally with metallographic paper. Smooth specimens for the rotating-beam tests were machined to approximately the desired diameter using successively smaller cuts; they were then polished with No. 000 metallographic paper, the final polishing being in the longitudinal direction. For the notched-type specimens, the notches were cut with a carefully ground tool of very fine-grained high-speed steel. The radius at the root of the notch was about 0.0002 in., as illustrated in Fig. 2.

Tests of each material were made using specimens with and without the anodic treatments; the treatments were applied to the specimens after all machining was completed. Photomicrographs illustrating the two extremes of thickness of coatings investigated in the tests of 75S-T6 are shown in Figs. 3 and 4.

In Figs. 5 to 10, inclusive, the results of the fatigue tests of the uncoated specimens are represented by open symbols, and smooth curves have been drawn through these points. The results of the tests of coated specimens are represented by solid symbols.

DISCUSSION OF RESULTS

The results of the tests of the 17S-T3 alloy sheet are shown in Fig. 5. The data show no definite effect of the Alumilite 205 treatment, except possibly at stresses below 24,000 psi., where the treatment may be slightly beneficial.

The anodic treatment used in the tests of the 17S-T4 alloy rod was the same as in the tests of 17S-T3 alloy sheet. As shown in Fig. 6, similar results were obtained in that the effect, if any, of the treatment was slightly beneficial.

In the tests of the 25S-T6 alloy-forged slab, the thicker anodic coating produced by the Alumilite Black 100 treatment was used. As shown in Fig. 7, this coating had no definite effect on the endurance limit (15,000 psi., based on 500,000,000 cycles of stress), but at stresses of about 17,000 psi. or higher, the coating definitely decreased the fatigue life. At one million cycles the decrease in fatigue strength was about 25 per cent. Apparently, the coating did not have sufficient ductility to withstand cracking at the higher stresses.

Two coatings having thicknesses of about 0.0001 and 0.0005 in., respectively, were used in the tests of 75S-T6 alloy rod and extruded bar. As shown by the data from the tests of smooth specimens in Figs. 8 and 9, the thinner coating produced by the Alumilite 1224 treatment had a slightly beneficial effect, while the thicker coating produced by the Alumilite 204 treatment had a slightly harmful effect.

These effects of the Alumilite 204 treatment on 75S-T6 alloy were observed throughout the entire range of stresses at which tests were made. While this differs in one respect from the results of the tests of 25S-T6 alloy with the Alumilite Black 100 treatment, where there was no harmful effect at stresses near the endurance limit of 25S-T6 alloy, it will be noted that the stresses in the tests of 75S-T6 alloy exceeded the minimum stress at which there were harmful effects on the fatigue strength of the 25S-T6 alloy.

The tests of 75S-T6 alloy rod containing a sharp V-notch indicated, as can be seen in Fig. 8, that neither of these two coatings had any definite effect on the fatigue strength in the presence of severe stress concentrations. As can be seen from a comparison of the photographs in Figs. 2 and 4, the Alumilite 204 treatment tended to increase the radius at the root of the notch. Any detrimental effect of the coating then probably was offset by this increase in notch radius. Since machine and structural parts seldom are free from stress concentrations, the lack of any effect of these coatings in the tests of notched specimens seems even more significant than the small effects observed in the tests of smooth specimens.

The results of the tests of B355-T6 alloy sand-cast bars are plotted in Fig. 10. As in the tests of the wrought alloys with similarly thin coatings, they show no definite effect of the Alumilite 704 treatment.

CONCLUSIONS

From the fatigue tests reported herein, involving complete reversal of stress, the following conclusions regarding the effects of anodic coatings on the fatigue strengths of aluminum alloys can be drawn:

1. Thin anodic coatings (about 0.0001 to 0.0002 in. thick) on smooth surfaces have little, if any, effect on the fatigue strengths of aluminum alloys. Such effects as exist seem to be beneficial rather than detrimental.
2. Thick anodic coatings (about 0.0005 to 0.0010 in. thick) on smooth surfaces seem to have slightly detrimental effects at higher stresses, but no shortening of fatigue life has been noted at stresses below 17,000 psi.
3. The findings of other investigators support the foregoing conclusions.
4. Since anodic coatings 0.0001 and 0.0005 in. thick were found to have no harmful effect on the fatigue strength of 75S-T6 alloy specimens with sharp notches machined in them before anodizing, it appears that aluminum alloys in the presence of severe stress raisers are not adversely affected by anodic coatings.

Acknowledgment:

The authors are grateful to Aluminum Company of America for permission to publish the data presented herein, to those members of the staff of the Aluminum Research Laboratories who conducted the tests, and to R. L. Templin, Chief Engineer of Tests and Assistant Director of Research, under whose general direction the work was performed.

DISCUSSION

Mr. Leo Schapiro.[1]—I recall that a paper on a similar subject was presented before a meeting of the Society of Automotive Engineers in 1949 by Messrs. R. C. Henshaw, L. Wallerstein, and S. J. Zand entitled "Fatigue Life of Aircraft Engine Mounting Components." In this paper, however, the emphasis seemed to be on the type of coating, that is, whether sulfuric or chromic acid; in the present paper the emphasis seems to be on thickness. I should appreciate having the authors' comments on this.

Messrs. G. W. Stickley and F. M. Howell (*authors' closure*).—The paper to which Mr. Schapiro referred included the results of comparative tests of some 75S-T6 aluminum alloy links that had been given anodic coatings using the sulfuric and chromic acid processes. Higher fatigue strengths were obtained when the chromic acid process was used. While the authors did seem to put the emphasis on the type of coating, rather than the thickness of coating, they also stated that "Anodic films formed in sulfuric acid are much thicker than those formed in chromic acid," the difference being in the order of 10 to 1. They also noted that the differences in behavior of coatings produced by the two processes are not ". . . due to differences in chemical composition or molecular aggregation."

The evidence seems to indicate that differences in thickness of coating are a much more important factor than the type of electrolyte. To substantiate this, tests similar to those described in our paper are being made using both thin and thick coatings produced by each process. When these studies are completed, we hope to present the results in another paper.

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[1] Chief Metallurgist, Materials Lab., Douglas Aircraft Co., Inc., Santa Monica, Calif.

EFFECT OF VARIOUS TREATMENTS ON THE FATIGUE STRENGTH OF NOTCHED S-816 AND TIMKEN 16-25-6 ALLOYS AT ELEVATED TEMPERATURES*

By W. E. Jones, Jr.[1] and G. B. Wilkes, Jr.[1]

Synopsis

A rather extensive program has been undertaken to determine the fatigue strength of notched specimens of S-816 (a cobalt-base alloy) and Timken 16-25-6 (a highly alloyed iron-base alloy) from room temperature to 1200 F. Various corrective treatments were tried, such as shot peening, coining, and heat treating, to improve the notched strength.

Ground notches tend to contain tensile stresses to a degree depending upon the severity of grinding. Shot peening overcomes these stresses and introduces compressive stresses, improving the fatigue strength. On the alloys tested there is additional improvement in strength by the cold work applied by shot peening. Alloy S-816 was tested with ground notches, and improvements in the order of 250 per cent were obtained at room temperature by shot peening, the effect diminishing to approximately 40 per cent at 1200 F.

Notches (in such materials) produced by machining do not have a high degree of tensile stress, in fact a compressive stress is indicated so that improvement by shot peening is of much less degree. Timken 16-25-6 was tested with machined notches; an improvement of 20 per cent was obtained at room temperature by shot peening, diminishing to essentially 0 per cent at 1200 F.

Several general conclusions can be drawn regarding notches and concerning treatments intended to reduce their damaging effect which should be of value to designers.

The object of this work was first to determine the fatigue strength of notched specimens of both S-816 and Timken 16-25-6 as compared with tests on smooth bars. This was done for temperatures from 75 F. to 1200 F. Assuming then that a part must be made with a notch, the second phase of the work was to devise corrective treatments that would improve the strength. It was desired that these improvements also be effective at elevated temperature and hence a large amount of elevated temperature testing was done.

Fatigue-Testing Equipment

Vibrating cantilever type machines were used for all tests and were of two different types: one pneumatically operated and the other electromagnetically operated. The pneumatically driven units have been described previously.[2] As a brief review, however, the general construction of the machine is shown in Fig. 1. It is of the vibrating cantilever type with vibration maintained (at resonance) by air supplied to a tuned air column. The basic principle of the driving

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Metallurgical Engineers, Thomson Laboratory, General Electric Co., West Lynn, Mass.

[2] F. P. Quinlan, "Pneumatic Fatigue Machines," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 846 (1946).

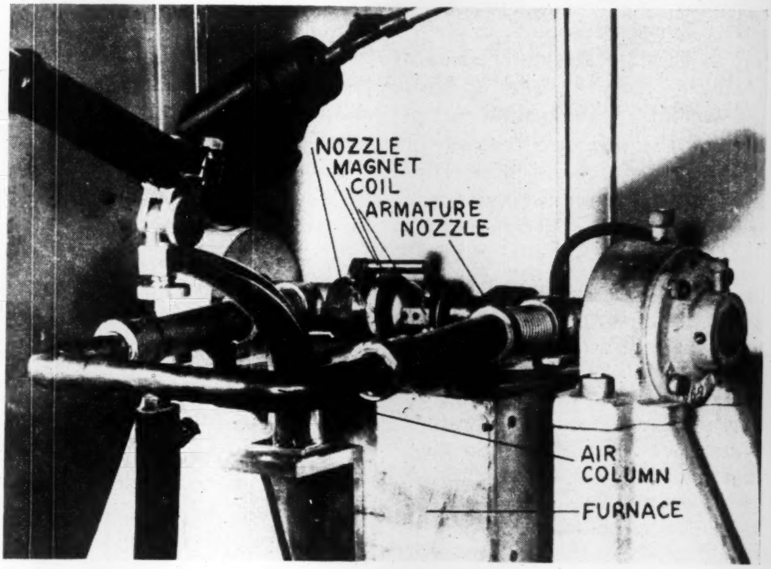
NOZZLE
MAGNET
COIL
ARMATURE
NOZZLE
AIR COLUMN
FURNACE

FIG. 1.—Pneumatic Fatigue Machine Used for Part of These Tests.

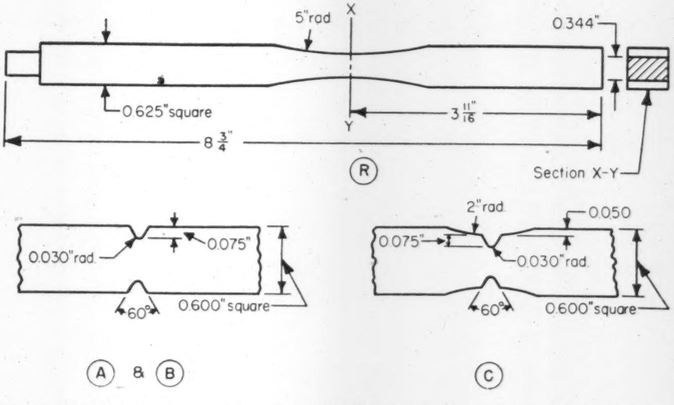
5"rad
X
Y
0.344"
0.625"square
3 11/16"
8 3/4"
R
Section X-Y
0.030"rad
0.075"
60°
0.600"square
A & B
2"rad
0.050
0.075"
0.030"rad
60°
0.600"square
C

FIG. 2.—Sketch of the Standard Test Bar and the Two Types of Notches Which Were Substituted for the Reduced Section in the Standard Bar.

mechanism is simply that as the piston moves away from one nozzle, the air velocity increases and the pressure drops while at the same time the other piston is closing the nozzle, reducing the velocity, and increasing the pressure. This unbalance of pressures in each half cycle of vibration is sufficient to maintain vibration. The electromagnetic machines are similar except for a longer armature and electromagnets driven by an oscillator replacing the air column. A small capacity pickup serves to match the oscillator frequency to that of the specimen and to control the amplitude. The frequency of test in both types of machines varies with the temperature and the bar, but was generally between 150 cps. and 200 cps.

The basic type of test bar and the various modifications is shown in Fig. 2. The standard bar is rectangular in cross-section (R in Fig. 2). The notched bars ((A, B, and C) Fig. 2) were of two types; the second, C, being used in the latter part of the program because of better vibrating characteristics. For convenience in the following discussion, the types of bars used are designated as follows:

Standard Bar.—R in Fig. 2—This is the standard test bar for the machines used and is assumed to have no stress concentration.

Notch A—A and B in Fig. 2—This is a ground notch finished in one pass and exhibits considerable evidence of overheating. To better describe the degree of overheating, the entire notch was ground in about 5 sec. without any coolant. Near the end of the cut there was a definite bright red color in the metal at the surface of the notch, which, combined with the character of the oxide built up, indicate a temperature of at least 1500 °F. The discoloration extended for about $\frac{1}{16}$ in. away from the base of the notch on the side of the bar. The theoretical stress concentration factor[3] is about 2.7.

Notch B—A and B in Fig. 2—This notch is the same as A in all respects except that it was ground with a first pass of 0.070 in. and a second finish pass of 0.005 in. The discoloration on the ground surface due to overheating in the first pass was removed in the second pass. Better dimensional tolerances and finish were also obtained.

Notch C—C in Fig. 2—This notch is identical to notch B except for the slightly reduced cross-section prior to notching. The theoretical stress concentration factor is about 2.4.

DEFINITION OF TERMS

Throughout this paper, unless otherwise stated, all reference to "fatigue results" refers to the fatigue strength at 10^8 cycles. The term "strength reduction factor" or the abbreviation "SRF" is defined as the ratio of the smooth bar strength to the notched bar strength at 10^8 cycles.[4] The strength reduction factors obtained are applicable only to the specific conditions of these tests. If used judiciously, however, the general conclusions may be applied qualitatively to other applications. All stresses were calculated from the formula $S = MC/I$ by the inertia method (see Appendix for details) where:

$$M = K \int_0^l \rho A f^2 dx$$

K = constant made up of conversion factors,
l = distance from notch to free end of bar,
ρ = lb. per linear inch of bar (a function of x depending on dimensions and density),
A = amplitude (a function of x),
f = frequency,
x = distance above notch,
C = distance from neutral axis to base of notch, and
I = moment of inertia of area between notches.

MATERIALS TESTED

The two alloys chosen for this program were a cobalt base alloy known as S-816 and an iron-base alloy, Timken 16-25-6. Their nominal analyses are as follows:

Analyses, per cent	S-816	Timken 16-25-6
Iron	3	52
Chromium	20	16
Nickel	20	25
Molybdenum	4	6.5
Tungsten	4	..
Cobalt	44	..
Manganese	1.5	1.2
Carbon	0.4	0.1
Columbium	4	..
Nitrogen	..	0.15
Silicon	0.75	0.75

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[3] G. H. Naugebauer, "Stress Concentration Factors and Their Effect on Design," *Product Engineering*, Vol. 14, February, 1943, pp. 82–87.

[4] "Symbols and Nomenclature for Fatigue Testing," ASTM BULLETIN, No. 153, August, 1948, p. 36; also "Manual on Fatigue," Am. Soc. Testing Mats., p. 3 (1949) (issued as separate publication, *STP No. 91*).

During the course of the testing, several heats of each analysis were used with reasonable correlation of results indicating that the results are typical of the alloy rather than of a specific heat.

The S-816 test bars were cut from $\frac{7}{8}$-in. round bar stock water quenched after 2150 F. for 1 hr. and then aged for 12 hr. at 1400 F. The material for the Timken 16-25-6 specimens was forged from a billet to a $1\frac{3}{4}$-in. thick slab, reheated to 1350 F. and cold worked 20 per cent, and stress relieved for 8 hr. at 1200 F. Smaller pieces from which to make the bars were then sawed from the slab.

RESULTS OF TEST

Results for S-816:

The basic problem with regard to S-816 was to determine the fatigue strength of a bar with a ground notch, in which the grinding was quite severe and then determine the degree of improvement obtained by various treatments. Figure 3 shows the normal smooth bar fatigue curve at 75 F., as well as two curves for untreated notched specimens. The lower curve is for a notch ground in one pass without coolant, with considerable overheating of the remaining metal (notch A). The middle curve is for the same notch ground very carefully with many passes, which showed no signs of any discoloration due to overheating. From these curves it can be seen that the possible loss in strength is rather large, the strength reduction factors being 4.6 for the single pass grind and 2.4 for the careful grind. This means that with the notch carefully ground the loss of strength due to the stress concentration is slightly less than that calculated by elastic theory, and therefore the material may be capable of partially relieving such a condition. It is obvious (and not surprising) that overheating during grinding is very damaging over and above the effect of the notch.

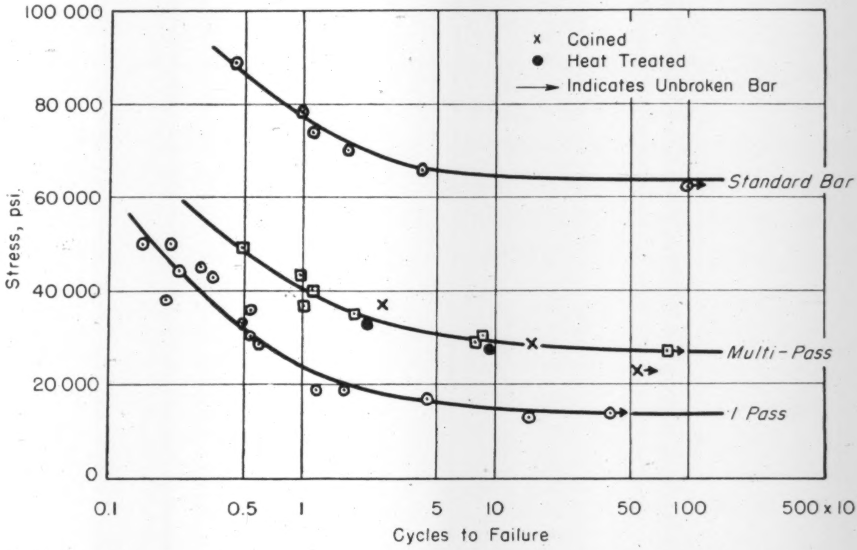

FIG. 3.—Fatigue Curves for S-816 at Room Temperature for Conditions Indicated.

A few specimens notched in one pass

(notch *A*) were coined and then tested at room temperature. The coining consisted of applying pressure to the radius at the base of the notch sufficient to upset the metal increasing the notch depth by 0.0015 in. The punch used to apply the pressure was shaped to fit the base of the notch exactly and was relieved on the sides so that the load was applied only in the radius at the base of the notch. A distinct improvement over the untreated notch is noted (Fig. 3). Since the coining amounted to severe cold work only at the very bottom of the notch, the point of failure was moved to the side of the notch beyond the coined area; the results are a consequence of coining but not the strength of coined material. The degree of improvement, therefore, is a function of the area on the sides of the notch that is coined until the true strength of the coined material at the base of the notch is reached.

A limited amount of data was obtained on notch *A* specimens which were given a complete heat treatment after notching (held at 2150 F. for $\frac{1}{2}$ hr.—water quenched, held at 1400 F. for 12 hr.—air cooled). This produced an essentially stress-free notch which could be used as a basis of comparison. The results (Fig. 3) fall very nearly on the curve for the carefully ground notch indicating that this also is essentially a stress-free notch.

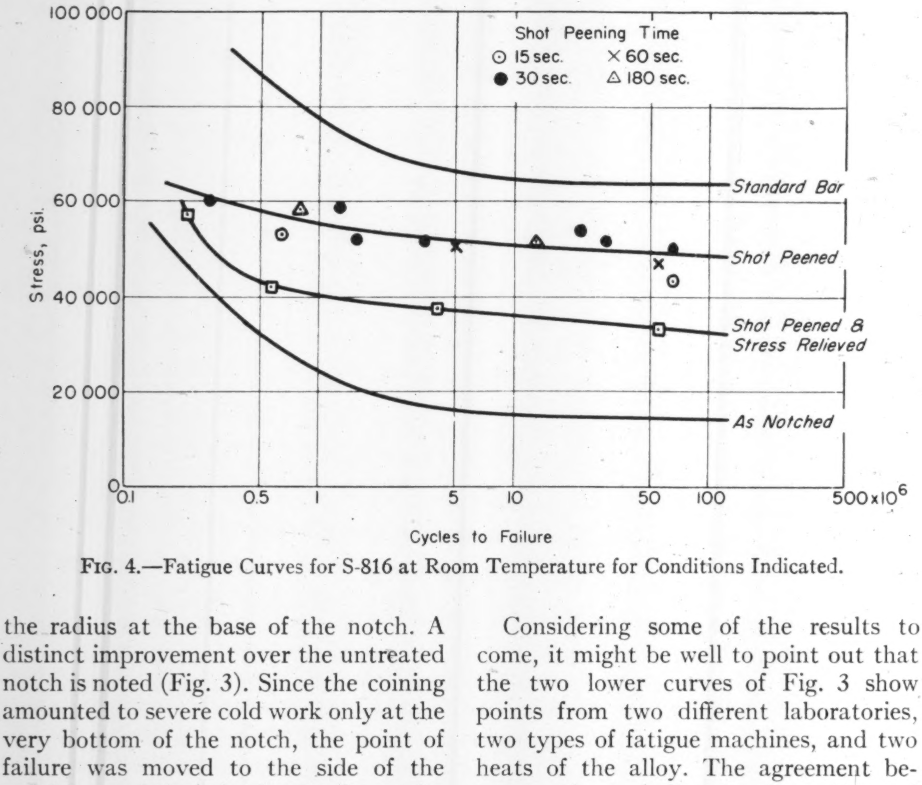

FIG. 4.—Fatigue Curves for S-816 at Room Temperature for Conditions Indicated.

Considering some of the results to come, it might be well to point out that the two lower curves of Fig. 3 show points from two different laboratories, two types of fatigue machines, and two heats of the alloy. The agreement between them indicates the order of accuracy of the testing equipment as well as the similarity of the heats.

The first attempt at a corrective treatment consisted of shot peening the surface of the notch before testing. (Details of shot peening procedure and its effects on the surface are covered in the Appendix.) In this case the notch was of the one pass or badly overheated variety (notch *A*), and the marked improvement obtained is shown on Fig. 4.

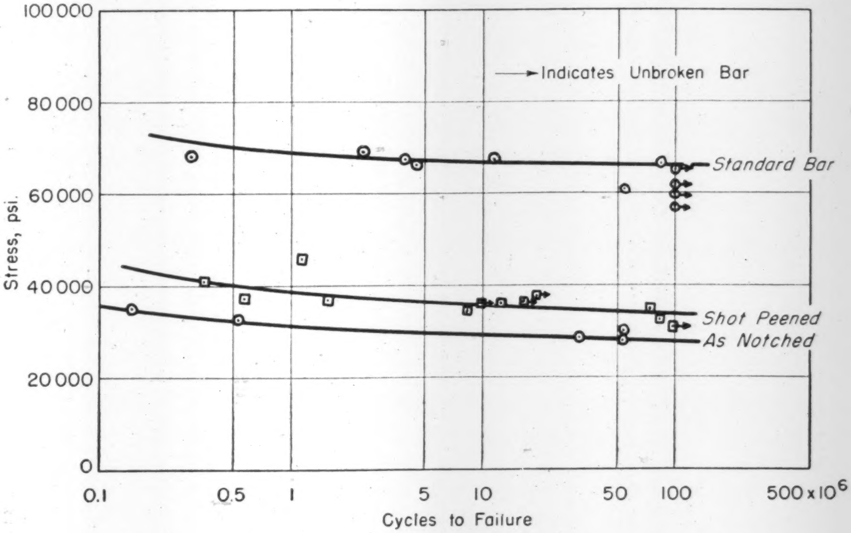

FIG. 5.—Fatigue Curves for S-816 at 1200 F. for Conditions Indicated.

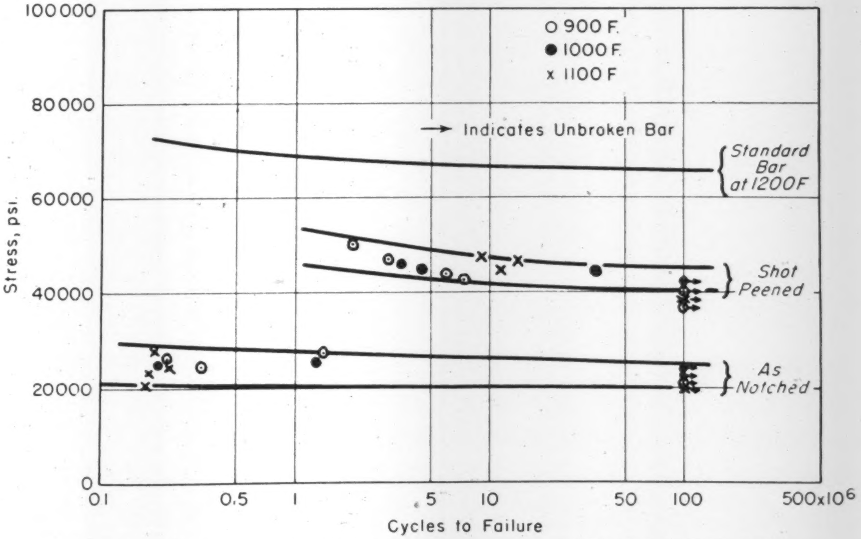

FIG. 6.—Fatigue Curves for S-816 at Temperatures and Conditions Indicated.

The SRF has been lowered from 4.6 to about 1.3. The shot peening was done for the times shown on the curve, 15 sec. being probably somewhat short and anything over 30 sec. of no advantage. The scatter in the results is apparently typical of notched specimens.

As an indication of the effect of temperature on the shot-peened notch, a test was run at room temperature on bars given a stress-relief anneal of 24 hr. at 1200 F. after shot peening (Fig. 4). The SRF was increased by the stress-relief treatment from 1.3 to 1.9, probably due to reduction of compressive stresses.

Complete tests were then run at 1200 F. The basic curves are shown in Fig. 5. The top curve is that for unnotched S-816. The lower curve indicates the notch effect, and the curve in between averages the somewhat scattered points from notched and shot-peened bars. By comparison with the room temperature tests, it can be seen that when tested at 1200 F., the effect of the one pass grind is much less severe and the effect of the shot peening is less beneficial. A possible explanation of this is the relief of internal surface stresses. The only reasons for an SRF greater than the theoretical stress concentration factor for the one-pass notch are actual cracks formed, structural change in material, or high surface tensile stresses. Since testing at 1200 F. raises the unpeened strength, it is unlikely that cracks were present and since the 1200 F. testing temperature is not high enough to cause any marked change in structure it must be assumed that the increase is due to the relief of the tensile stresses. By the same token the compressive stresses left by shot peening are somewhat relieved in testing at 1200 F. This relief of stresses would cause an increase in the notched unpeened curve and a decrease in the shot-peened curve as was observed.

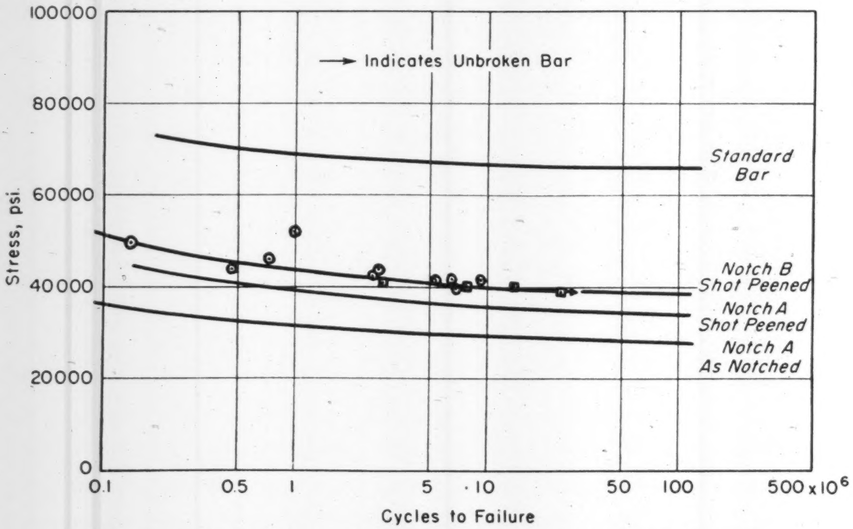

FIG. 7.—Fatigue Curves for S-816 at 1200 F. for Conditions Indicated.

Figure 6 shows the results of tests run at 900, 1000, and 1100 F. The scatter in the test points appears to be greater than the difference in strength due to test temperature, and they can only be discussed as a group. There is a marked improvement in the "as-notched" results lowering the SRF from 4.6 at room temperature to 2.9 in this temperature range as against 2.4 at 1200 F. At the same time there is some loss in the shot peened tests; the SRF rising from 1.3 at room temperature to 1.5.

At this point in the program a slightly

different notching practice was introduced in order to more nearly simulate a reasonable production procedure. The new notch (designated as notch *B*) was ground in two passes: one heavy pass of 0.070 in. which produced considerable overheating and one light pass of only 0.005 in. as a finishing cut. In all other respects the new notch was identical to the previous one. Results of 1200 F. tests on this new notch are depicted in Fig. 7. The SRF of the shot-peened notch has been reduced slightly from 1.9 to 1.7. This improvement is probably due mainly to the removal of the most overheated layer by the light finishing pass.

A third type of notch (notch *C*) was also used, the shift being made chiefly to obtain more stable vibrating characteristics. This notch was identical to notch *B* except for a radius cut in the bar prior to notchnig. The theoretical stress concentration factor, however, was about 10 per cent less, being approximately 2.4. The results at 1200 F. are plotted in Fig. 8. The results are similar to the same conditions for the other notches, small differences being due to the slightly different notch as well as a different heat.

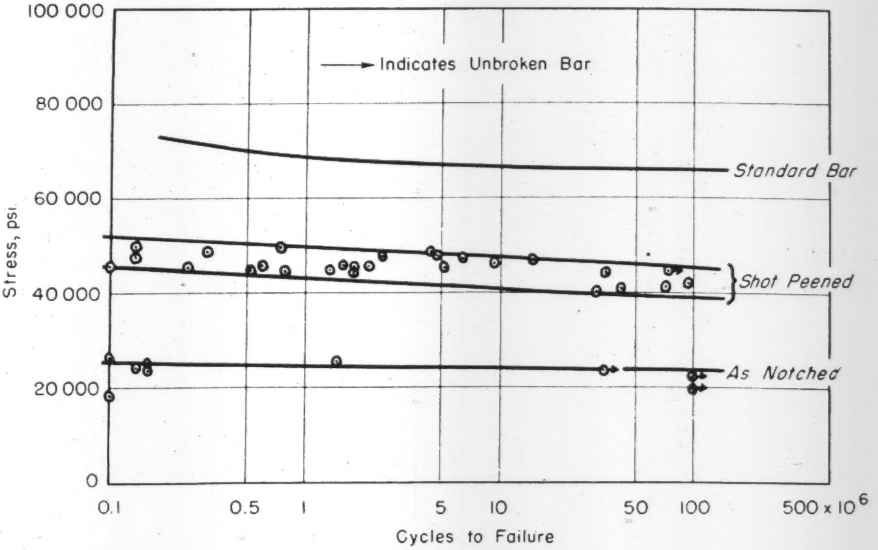

FIG. 8.—Fatigue Curves for S-816 at 1200 F. for Conditions Indicated.

TABLE I.—BASIC DATA FOR S-816.

Condition of Test	Strength Reduction Factor		
	75 F.	900 F. to 1100 F.	1200 F.
Notch A shot peened	1.3	1.5	1.9
Notch B shot peened	...	...	1.7
Notch C shot peened	...	...	1.6
Notch A shot peened—stress relieved at 1200 F.	1.9	...	...
Multi-pass grind	2.4	...	...
Notch A heat treated	2.5[a]	...	...
Notch A coined	2.4[a]	...	...
Notch A untreated	4.6	2.9	2.4
Notch C untreated			2.7

[a] Estimated values from only a few points.

Table I summarizes the basic data for S-816 on a strength reduction factor basis. It can be seen that at room temperature the detriment due to grinding and the benefits due to shot peening are maximum and that the effect of temperature is to reduce the extremes. If the trends continued as the test temperature is raised, it seems possible that shot peening would have no lasting effect or might even do damage. In fact, limited data[5] obtained at room temperature on specimens stress relieved at 1500 F. show the not shot peened to be superior to the shot peened specimen.

There are considerable data,[6, 7] as yet inconclusive, indicating that S-816 may be seriously damaged by prolonged vibration at stresses below the fatigue limit, manifested by much shorter than expected life if the stress is raised into the failure range. With this in mind, a series of tests was run on the notched test bars at 1200 F. designed to establish the effect of prestressing at a high level prior to running the fatigue test. The particular procedure for these tests called for vibrating the test bars at such a stress that one-third would fail at or before 3.5 × 10^6 cycles. Those bars that did not fail were then run at various stresses in order to obtain a fatigue curve on prestressed material. The object of this schedule was to show the effect of short periods of overload on the total life of any piece of apparatus wherein vibration may be a problem.

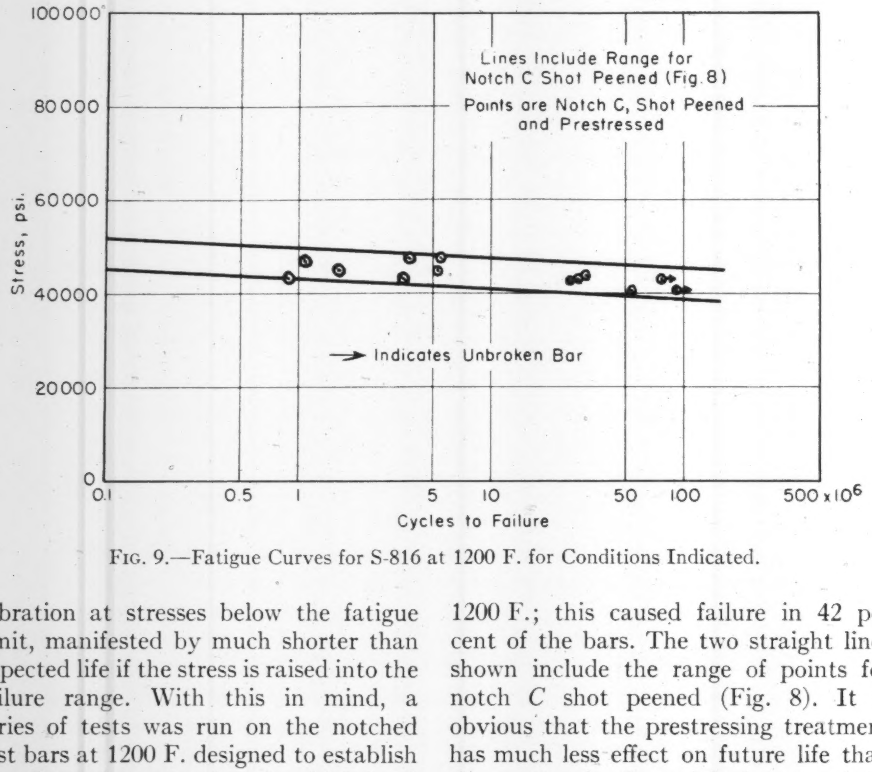

FIG. 9.—Fatigue Curves for S-816 at 1200 F. for Conditions Indicated.

Figure 9 shows the results (with notch *C* after shot peening) of prestressing at 45,000 psi. for 3.5 × 10^6 cycles at 1200 F.; this caused failure in 42 per cent of the bars. The two straight lines shown include the range of points for notch *C* shot peened (Fig. 8). It is obvious that the prestressing treatment has much less effect on future life than the scatter in the original test points. Two other prestressing tests were run, one with a 1200-F., 24-hr. stress-relief anneal interposed between the prestressing and the final test, and the other with reshot-peening interposed in the same manner. These two tests are shown in Figs. 10 and 11. On these curves the range of notch *C* shot peened is again shown by lines. Unfortunately, the scatter in the results completelys obscure any effect of prestressing or subsequent treatments, the only pertinent comment being that the prestressing did not have any large magnitude effect as compared to the scatter.

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[5] F. C. Robertshaw, General Electric Co., West Lynn, Mass., unpublished data.

[6] P. D. Gorsuch, General Electric Co., Schenectady, N. Y., unpublished data.

[7] G. B. Wilkes, Jr., General Electric Co., West Lynn, Mass., unpublished data.

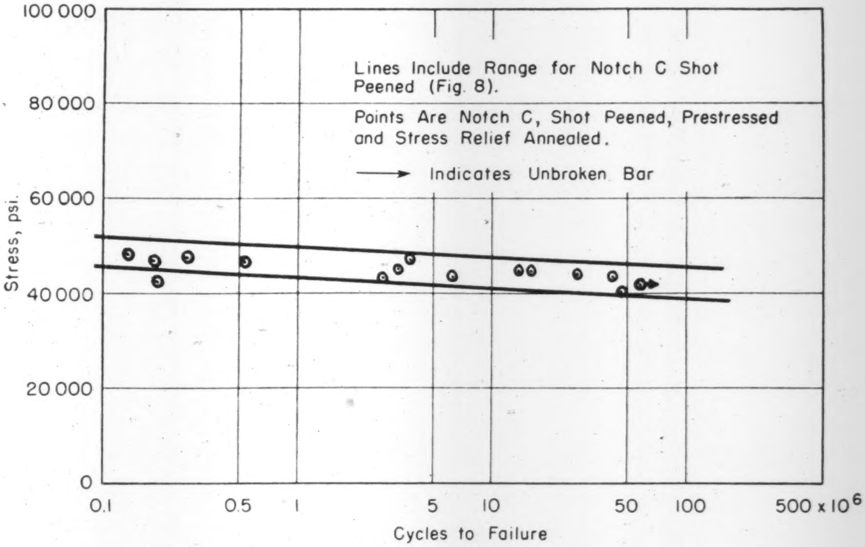

FIG. 10.—Fatigue Curves for S-816 at 1200 F. for Conditions Indicated.

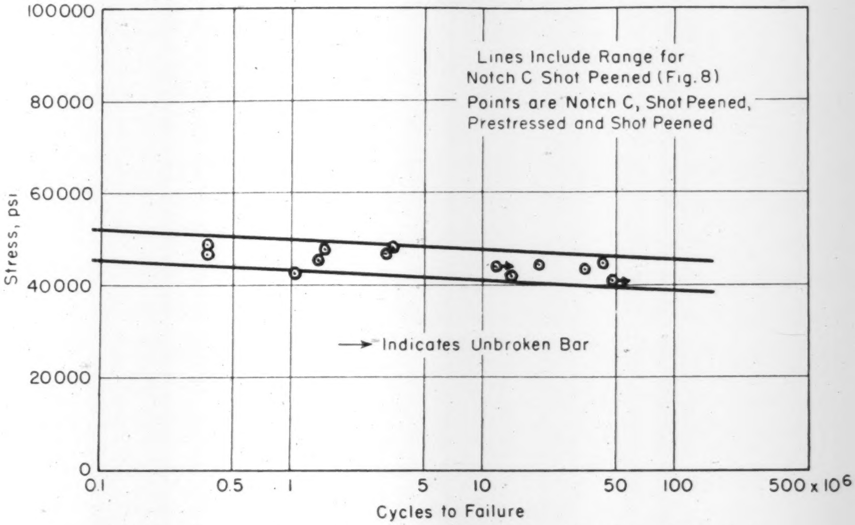

FIG. 11.—Fatigue Curves for S-816 at 1200 F. for Conditions Indicated.

Results for Timken 16-25-4:

A similar series of tests was run on Timken 16-25-6. In all cases the notch was identical in shape to notch *C* (Fig. 2), the only difference being that the notch was cut in a shaper instead of ground. This difference in manufacture changed the notch from one overheated and probably containing residual tensile stresses (as in the alloy S-816) to one cold worked and probably containing residual compressive stresses. Figure 12 shows the room temperature results. Expressing the notch fatigue strength in the same terms as for alloy S-816, the SRF is 1.8 for the "as-notched" specimens and 1.4 after shot peening. This indicates some advantage in shot peening

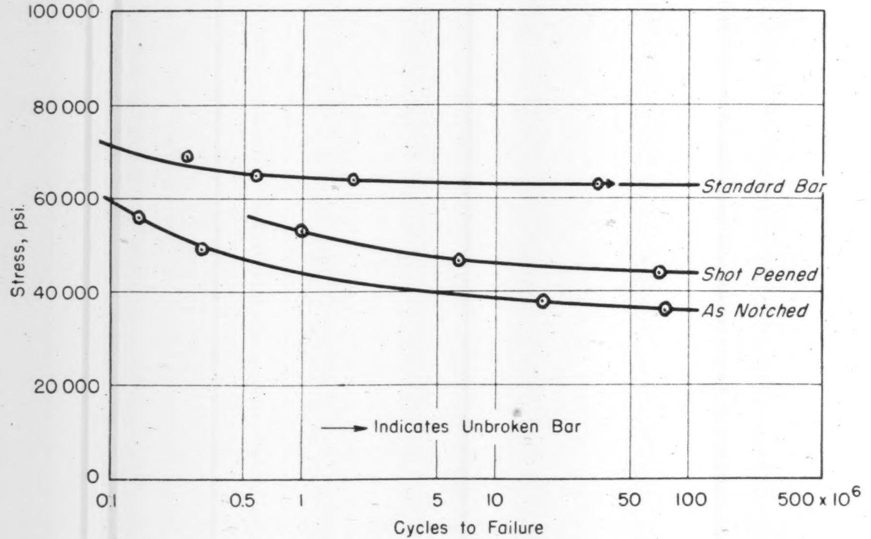

FIG. 12.—Fatigue Curves for Timken 16-25-6 at Room Temperature for Conditions Indicated.

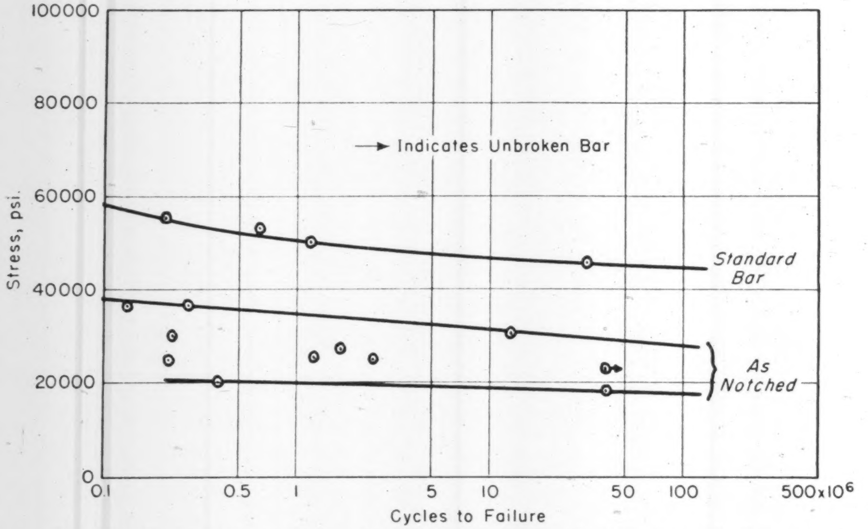

FIG. 13.—Fatigue Curves for Timken 16-25-6 at 1200 F. for Conditions Indicated.

but to a much less degree than for S-816 due to more favorable notch conditions.

The 1200-F. tests on "as-notched" specimens are shown in Fig. 13 along with the unnotched curve. The scatter prevents any but the most general conclusions. A new heat of material was introduced as a check of the shot-peened tests with no reduction in scatter, which leads to the conclusion that the scatter is inherent in notched Timken 16-25-6 specimens at 1200 F. rather than being due to faulty material. The SRF for these "as-notched" bars seems to be between 1.6 and 2.5, with an average somewhere in the neighborhood of 2.0. It should be pointed out that, whereas the untreated S-816 notch became stronger as the test temperature was raised, the untreated Timken 16-25-6 notch becomes weaker at higher temperatures. This is undoubtedly due to the relief of surface stresses which are undesirable tensile stresses in the S-816 and desirable compressive stresses in the Timken 16-25-6.

Turning now to the shot-peened tests on this material, shown in Fig. 14, the large scatter of points is still present. Here, again, only a rough estimate of results can be given; the SRF is in the range 1.5 to 2.0 with an average of 1.7. The apparent gain in strength by shot peening is small (2.0 to 1.7) and appears to have been obtained by strengthening the weakest bars with no change in the strong ones. This could be of more value than the change in SRF. Since these bars were cut from a larger piece that required cold work, which might vary from bar to bar, to develop its properties, a hardness check was made on each bar. The readings varied over the range of 250 to 321 Brinnel with absolutely no correlation with the fatigue strength.

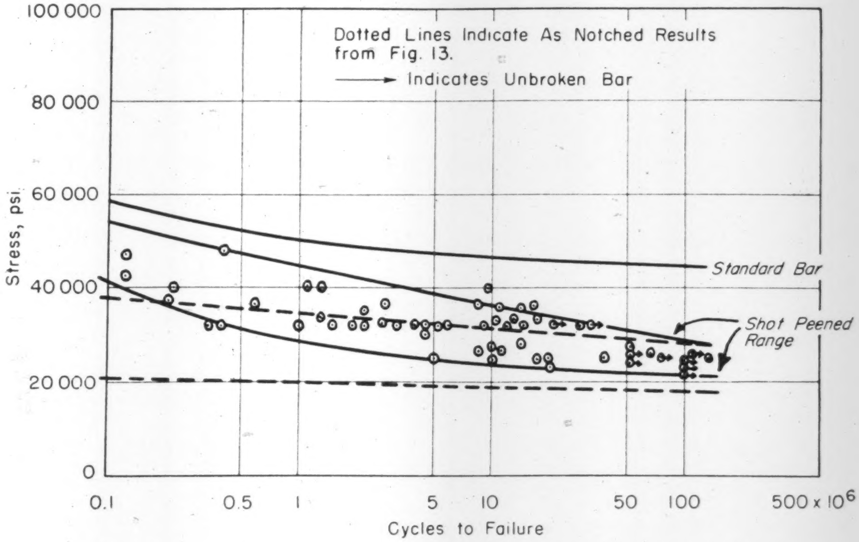

FIG. 14.—Fatigue Curves for Timken 16-25-6 at 1200 F. for Conditions Indicated.

Prestressing Timken 16-25-6 in the same manner as for S-816 gave much the same results. Prestressing the notched and shot peened bars and then testing with no intermediate treatment shows the same spread and the same average value as without the prestressing (Fig. 15). With an intermediate treatment (after prestressing) of 1200 F. for 24 hr., the spread and 10^8 cycle results are not changed, but the short cycle failures (up to about 5×10^6 cycles) appear weaker; in fact, the average of the test points is very nearly a horizontal line as seen in Fig. 16. When notched and shot-peened bars are prestressed and then shot peened again, the results are essentially

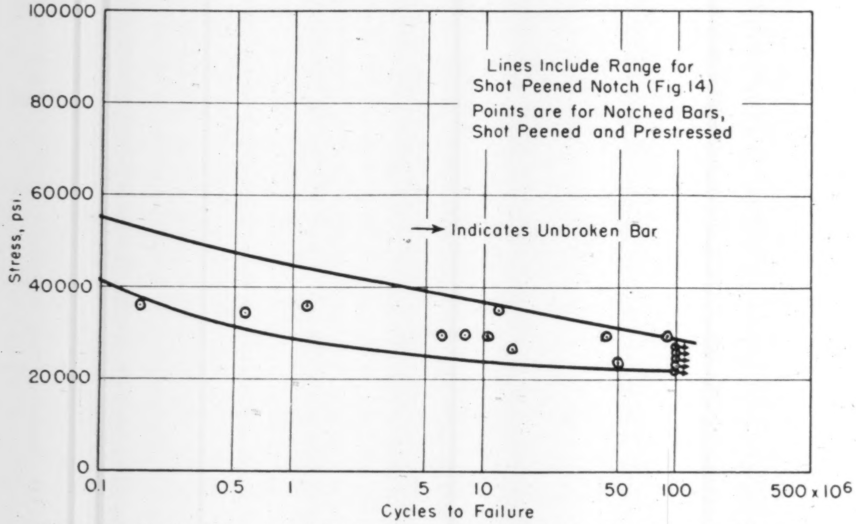

Fig. 15.—Fatigue Curves for Timken 16-25-6 at 1200 F. for Conditions Indicated.

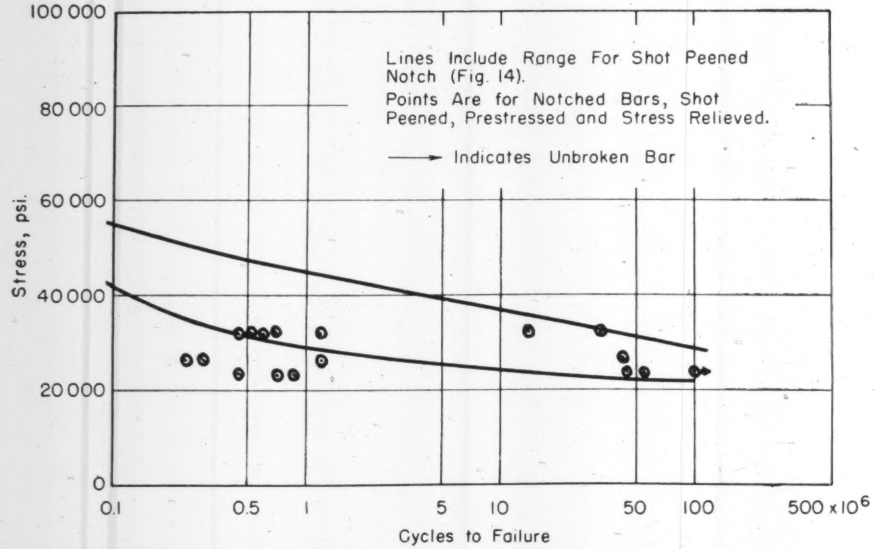

Fig. 16.—Fatigue Curves for Timken 16-25-6 at 1200 F. for Conditions Indicated.

the same as with no prestressing. The results are shown in Fig. 17.

The following table summarizes the basic Timken 16-25-6 results. In the case of the 1200-F. results, the range as well as the average is shown because of the scatter.

Condition	Strength Reduction Factor	
	75 F.	1200 F.
As notched	1.8	2.0 (1.6 to 2.5)
Notched and shot peened	1.4	1.7 (1.6 to 2.0)

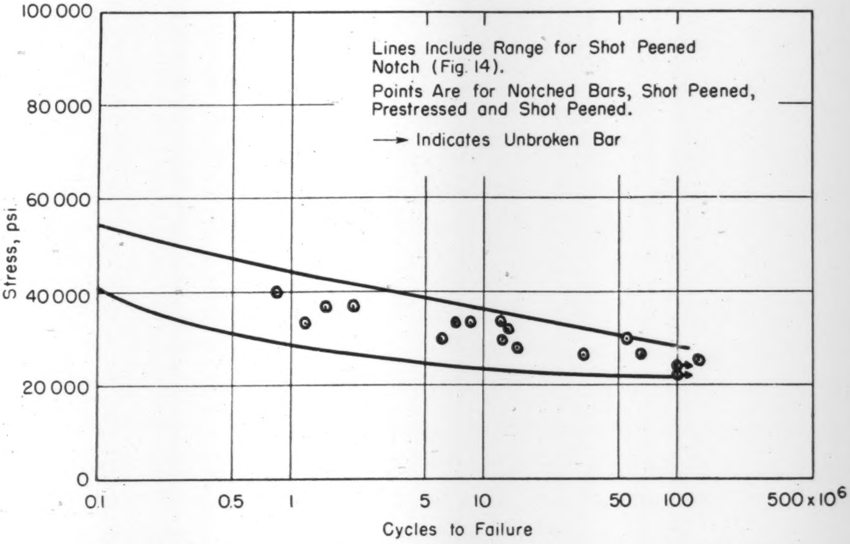

FIG. 17.—Fatigue Curves for Timken 16-25-6 at 1200 F. for Conditions Indicated.

It is immediately apparent that the machined notch is not nearly as severe as the ground notch, leaving much less room for improvement due to shot peening. The room temperature factors show the gain for shot peening. At 1200 F., however, there appears to be only a very slight gain (by strengthening weak bars) which is of small importance in the light of the excessive scatter of the test points. The prestressing tests, with or without additional treatment, show no apparent effect, indicating only that the effect is small compared to the spread in the results themselves.

CONCLUSIONS

While the results of these tests apply directly to the specific shapes and conditions used, there are several broad conclusions that can be drawn from various aspects of the work. There is no reason to believe that these broad conclusions would not be applicable to materials in general.

Notches:

It is a well-founded fact that notches, or other stress raisers, are not good practice for any application including one subjected to fatigue. The tests reported here indicate that there may be a large difference in the behavior of a given notch depending on how it is made. In the case of the notch ground in one pass accompanied by considerable overheating, the strength was only half as much as theory[2] predicted. Since suitable treatment could repair some of this damage, actual cracks could not have been a factor, and it appears logical that high residual tensile stresses at the base of the notch were an important factor. If, however, care is used in the grinding, these stresses are not set up and the theoretical stress concentration is obtained. On the other hand, if the notch is machined, particularly where the material is not too easily cut, apparently some compressive stresses are left which tend to enhance the fatigue strength.

The elevated temperature tests serve the dual purpose of (1) supplying the actual fatigue strength and (2) when compared with the room temperature tests giving an important clue to the state of stress at room temperature inasmuch as stress relief takes place under these conditions. The ground notch for example, becomes stronger (SRF 5.0 to 2.4) when tested at elevated temperature and the machined notch becomes weaker (SRF 1.8 to 2.0).

Corrective Treatments:

Assuming that a part requires a notch that will be subject to fatigue, there are two ways, shown by this work, to improve the strength. The best way is cold working the surface of the notch by shot peening, coining, rolling, etc., in such a way as to set up compressive stresses in the surface. This increases the strength in its own right, as well as helping to correct any tensile stresses that may have been produced in making the notch. If putting the surface of the notch in compression is not practical, a stress relief anneal or complete heat treatment will remove a large part of any tensile stresses that may be present. If compressive stresses are present, the part should not be annealed, as they are beneficial. If the part in question is to operate at elevated temperatures, the gain realized by cold working treatments becomes less as the temperature is increased, probably becoming negligible at the higher stress-relieving temperatures.

APPENDIX

Details of Shot Peening Procedure:

Shot size—S.A.E. S110 (average diameter 0.018 in.)
Pressure—to give arc height on Almen "A" specimen of 0.010 to 0.011 in. (in the order of 80 psi.)
Nozzle—$\frac{3}{8}$ in. suction type
Nozzle to work distance—2 in.
Peening time—30 sec. each side (unless otherwise specified)

It might be well to state that the term "shot peening" refers to the use of round metallic shot, as contrasted to "grit blasting" using angular metallic particles or "sand blasting" using sand.

The effect of this treatment on S-816 is shown in two photomicrographs, Figs. 18 and 19. Figure 18 shows the depth of the cold work imparted by shot peening as a recrystalized area. This was produced by solution-treating the worked surface 15 min. at 2050 F. and then aging 4 hr. at 1400 F. The average depth of effect is in the range of 0.002 in. to 0.003 in. The surface of a specimen after being vibrated at 1200 F. for 100 hr. is pictured in Fig. 19. In this case the depth of heavy precipitation is an indication of the shot-peening effect. The particular area shown in Fig. 18 was relatively unstressed since it was on the side of the notch. A similar specimen was examined after only 25 hr. of vibration at 1200 F. The degree of precipitation was much less except at the base of the notch where the high stress accelerated the action.

Timken 16-25-6 did not satisfactorily respond to the recrystalizing treatment, probably because of the 20 per cent cold work used in manufacture of the bars. Precipitation occurs, as in S-816, with time at 1200 F. Figure 20 shows the precipitation that occurred in 93 hr. at 1200 F. on the shot-peened surface.

A number of the specimens were checked for dimensional change caused by shot peening. This was done by placing a 0.100-in.

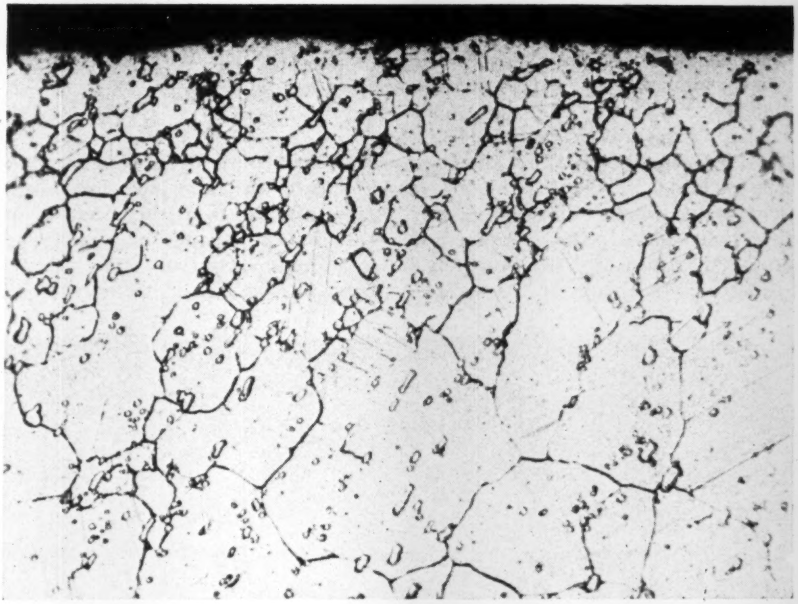

FIG. 18.—Photomicrograph of Surface of S-816 Fatigue Test Specimen After Shot Peening and Heat Treating to Recrystallize the Worked Area (×500).

Effect penetrates to about 0.002 to 0.003 in.

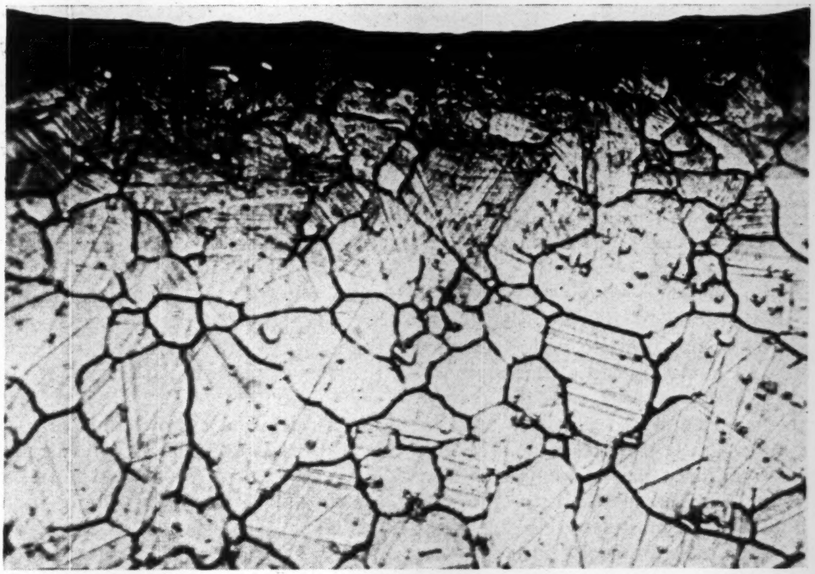

FIG. 19.—Photomicrograph of Surface of S-816 Fatigue Test Specimen After Shot Peening and Testing at 1200 F. for 100 hr. (×500).

Precipitation at surface is another measure of depth of effect of shot peening—0.002 to 0.003 in.

diameter pin in the two opposite notches and measuring the distance between the outside of the pins. After shot peening, the same measurement was made using the same pins the difference in distance being due to the shot peening. Changes of between 0.0003 in. and 0.0008 in. were obtained in a direction indicating expansion. The actual motion of the surface perpendicualr to its plane is only $\frac{1}{4}$ of these values, since in the above method two surfaces were measured and at an angle of 30 deg. from the plane of the surface.

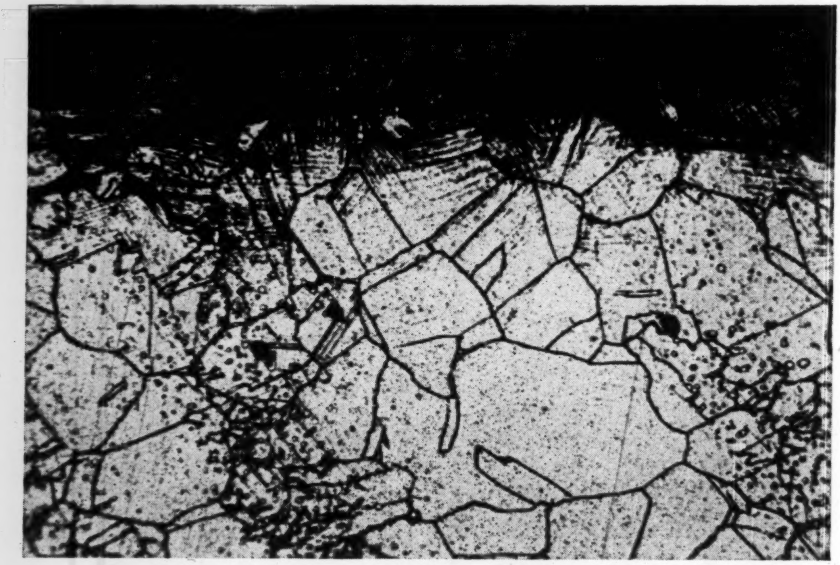

FIG. 20.—Photomicrographs of Timken 16-25-6 Fatigue Test Specimen After Shot Peening and Testing for 93 hr. at 1200 F. (×500).

The precipitation at the surface shows the depth of effect of shot peening to be 0.002 to 0.003 in.

Surface roughness measurements were made at a magnification of 100 and showed an average maximum peak to valley distance of 0.001 in. Comparing this to the pin measurements it can be seen that the average surface was compressed slightly, but the peaks raised are above the original surface level. It is obvious that the effects of shot peening (performed as stated) are well within all but the most exacting tolerances unless an extremely smooth surface is required.

Stress Calculations:

A graphical solution of the formula

$$M = K\int_0^l \rho A f^2 dx$$

was obtained by the following procedure: (1) Calculate the curve of weight distribution *versus* the length of the specimen from the bar and armature dimensions and their densities. (2) Experimentally determine the curve of deflection *versus* length of specimen when vibrating. (3) Calculate the curve of acceleration *versus* length corresponding to the measured deflection, the frequency, and the peak to peak displacement. (4) Multiply the weight distribution curve ordinates by the acceleration curve ordinates to obtain force distribution curve. (5) Integrate the elements of force (product of force per unit length and increment of length) multiplied by their respective moment arms about the notch. This gives M for insertion in the formula $S = MC/I$

DISCUSSION

MR. MARTIN FLEISCHMANN[1] (*presented in written form*).—Messrs. Jones and Wilkes presented a paper which should be of most timely interest to the designer of turbo-superchargers, jet engines, and gas turbines. While considerable data have been published on the high-temperature creep and stress-rupture properties of most "super alloys," information in regard to their fatigue strength at elevated temperatures is very limited.

Futhermore, little or no data had been available on the effect of notches on the fatigue strength at elevated temperature, and the authors should be highly commended for this original work which we hope will give some impetus for further investigation.

While valuable information was obtained on the two alloys tested, the S-816 and the Timken 16-25-6, a direct comparison of the properties should only be made after due consideration is given to the fact that the specimens of the S-816 material were cut from $\frac{7}{8}$-in. round bar stock while the test bars of 16-25-6 alloy were machined from forged slabs. No doubt this was done intentionally since the S-816 alloy is commonly used for turbine buckets while the Timken 16-25-6 material finds its most extensive application in turbine wheels.

We wonder, however, if the relatively large scatter of points reported for the 16-25-6 alloy may not be due to the characteristics of the slab, that is variations in grain flow and cold reductions in different sections of the slab from which the samples were taken. It should be of interest to obtain test results from cold-worked bars of 16-25-6 material which could then be compared directly with the data obtained on the S-816 alloy.

MR. P. R. TOOLIN.[2]– The authors deserve to be complimented on their excellent paper which adds considerably to our still scant knowledge of the fatigue properties of high-temperature alloys. It is felt that additional information on a few points would further increase the value of this paper.

Any available information on the hardness and on the tensile properties of the alloys would certainly be of interest.

The preparation of the standard specimens is not given, and while it is assumed that the 16-25-6 specimens were machined, an indication of the technique employed on the S-816 specimens would be worth while. It would also be of interest to know whether the corners of the standard specimens and the ends of the root of the notches were left sharp or were "broken" and if broken, to approximately what radius this was done.

The question naturally arises as to whether the fractures started, in general, on the face of the specimens or, as is frequently the case for rectangular specimens, at the corners.

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[1] **Metallurgical Engineer, The Timken Roller Bearing Co., Canton, Ohio.**

[2] **Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa.**

Perhaps it should be noted that while the authors' source for the (theoretical) stress concentration factors gives values of 2.7 and 2.4 for notches *A* and *C* respectively, if these factors are calculated by Neuber's derivation,[3] values of 2.4 and 2.2 are obtained. If these values are used, both the carefully ground notches and the ground and subsequently heat-treated notches in the S-816 specimens tested at room temperature give fatigue-notch factors (SRF) equal to the stress concentration factor, or a notch sensitivity, q,[4] of one. As this is a rather unusual occurrence (at least we have not observed such large notch sensitivities for the high-temperature alloys we have tested with round specimens), one wonders if the corner effect at the end of the notch may not be more severe for the notched specimen than for the unnotched specimen.[5] Similar tests on round specimens would be valuable as, while much of the current fatigue work throughout the country is being done on round specimens, the common turbine blade root designs have rectangular cross-sections.

MR. W. E. JONES (*author's closure*).—In answer to Mr. Fleischmann's discussion, no tests similar to those described in this paper were made on cold-worked Timken 16-25-6 bars. However, the bars tested were cut from forged and cold-worked slabs which would closely approximate bar stock.

Mr. Toolin's comments on this paper are appreciated. The method of preparation of test specimens is described in the paper. All test specimens were machined, and then the notch was ground on S-816 and machined on the Timken 16-25-6. The corners on the bars were broken but the ends of the root of the notch were not.

The Timken 16-25-6 ranged in hardness from 252 to 321 Brinell. Due to other variables, there was no apparent correlation between individual hardness values and the test results. The S-816 bars were made from production material which ranges in hardness from approximately 25 to 32 Rockwell C scale. Several heats of each material were used during the course of the tests.

Examination of the fractures indicated that there was no single area in which initial failure occurred. Some failures occurred at or near the corner, but many started on the face of the notch. Although there were some corner failures, there is no evidence in this data that there is any marked corner effect.

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[3] G. Neuber, "Kerbspannungslehre," Julius Springer, Berlin (1937); Translated by David Taylor Model Basin, J. W. Edwards (1946).

[4] $q = \frac{K_f - 1}{K_t - 1}$, where K_f equals the fatigue notch factor and K_t equals the (theoretical) stress concentration factor.

[5] T. J. Dolan, "Certain Mechanical Strength Properties of Aluminum Alloys 25S-T and X76S-T," *Technical Note 914*, issued in 1943 by Nat. Advisory Committee Aeronautics, gives data for round and rectangular machined specimens of 25S-T whichshows little variation in q between the two types of specimens.

WELDING CHARACTERISTICS OF OPEN HEARTH AND BESSEMER SEAMLESS STEEL PIPE*

By A. B. Wilder,[1] W. B. Kennedy,[1] and F. W. Crouch[1]

Synopsis

This paper discusses the welding characteristics of A.S.T.M. Specification A 53 grade B seamless steel pipe[2] and in particular the properties which are related to deoxidation practice and steel-making process. Deoxidized acid-bessemer, silicon-killed and silicon-aluminum-killed basic open-hearth pipe in the as-rolled condition were investigated. The deoxidized acid-bessemer steel was made by a process which is distinctly different from that used for ordinary bessemer steel.

The properties of girth welds made by a pipe fabricator and laboratory bead welds were studied. Tension and bend tests were made on the girth welds. A new type of weldability test, the weld bead flattening test for tubular products, is described. The internal structure and hardness of the girth and bead welds and the advantages of multipass welding are discussed. Impact properties of the girth welds were thoroughly investigated.

The material investigated met welding requirements of the A.S.M.E. Boiler Construction Code. For bessemer seamless pipe, lower hardenability, lower bead weld hardness and less cracking susceptibility was observed when compared with the open-hearth pipe. Bessemer and silicon-aluminum-killed open-hearth seamless pipe had higher impact properties at low temperature, less martensite and underbead cracking in the small weld bead and lower hardenability than silicon-killed open-hearth pipe.

Industrial piping systems, such as in the construction of power stations, oil refineries, and chemical plants, usually require special fabrication, and seamless pipe permits the maximum flexibility in piping design. Pipe fabricators make hot and cold bends, girth welded joints, and perform many other operations which are related to properties of the material. In the present investigation, the types of material to be discussed have been widely used by pipe fabricators and meet their requirements.

The pressures and temperatures utilized in industrial piping systems have progressively increased, and, as a result, attention has been focused on the design of piping systems and, in particular, the design of joints. There are in service today flanged and bolted joints, also screwed fittings, but joints of these types are generally costly for high temperatures and pressures. In addition to piping systems operating under high

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

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[2] Standard Specifications for Welded and Seamless Steel Pipe (A53 - 47), 1949 Book of A.S.T.M. Standards, Part 1, p. 7.

temperatures and pressures, there are piping systems with moderate pressures and temperatures where tight joints are desirable. The continued and even more general acceptance, therefore, of welding as a reliable method of joining pipe will be experienced in the future.

In the use of carbon steel seamless pipe for industrial piping, usually A.S.T.M. Specification A 53 grade B material[2] is employed, although in many applications A.S.T.M. Specification A 106[3] grade B material may be used. In the construction of transcontinental gas or oil pipe lines, large quantities of grade B material made in accordance with the American Petroleum Institute specifications for line pipe are also employed. Grade B material has a 35,000 psi. minimum yield strength and 60,000 psi. minimum tensile strength. Preheat is generally not employed in welding grade B seamless pipe as the chemical composition seldom exceeds 0.30 per cent carbon and 1.00 per cent manganese. Until recently only open-hearth steel was used in the manufacture of seamless pipe. However, in 1937 when the manufacture of lap-welded pipe from bessemer steel was discontinued by the National Tube Co., a new type (**1**)[4] of seamless pipe was developed. This new material was made from deoxidized acid-bessemer steel and has been included in A.S.T.M. Specifications A 53 since 1943. Ordinary bessemer steel is not killed or deoxidized and, therefore, has not been successfully used in the commercial production of seamless pipe.

Steel-Making Practice:

Ingots with a dense homogeneous structure, free from harmful porosity or blowholes, are produced from killed steel. It is this type of steel with a uniform composition which is usually employed in the manufacture of seamless pipe. In the production of killed open-hearth steel, careful control of the furnace charge, working the heat, slag practice and finishing the heat are necessary. During pouring of the steel into the ladle, strong deoxidizers such as ferrosilicon or aluminum are added. During teeming, no additions are made and the steel lies quietly in the mold as solidification takes place.

In the production of acid-bessemer steel for seamless pipe, a completely deoxidized steel is also used. It is this feature of the process which is responsible for the fact that deoxidized or killed bessemer steel for seamless pipe differs from regular acid-bessemer steel for butt or lap welded pipe. Production of seamless pipe is a difficult forging operation. A billet of uniform density and forging characteristics is required. Satisfactory seamless pipe has rarely been produced with uniform practice from either rimmed or capped steels. Since the introduction of bessemer seamless pipe, over 1,000,000 tons have been produced in various grades of tubular products.

Factors responsible for superior quality of deoxidized bessemer steel do not originate entirely from the fact that a killed steel is involved, but more precisely in the method of killing the steel When ordinary bessemer steel is made, carbon, manganese and silicon are removed during the blow, and ferromanganese is added to the ladle. A small amount of aluminum is added to the mold. Satisfactory seamless pipe has rarely been produced with uniform practice from this type of steel. In the manufacture of deoxidized acid bessemer steel, molten iron containing about 4 per cent carbon is added to the vessel at the end of the blow, and oxygen is removed from the molten metal as carbon monoxide. This addition of hot metal also adjusts the carbon content of the steel to meet specification requirements. The removal of oxygen as a gas rather than by combination with silicon or a similar deoxidizer to form inclusions improves cleanliness of the steel. This helps to provide a steel which may be satisfactorily pierced on a commercial basis. After carbon deoxidation, manganese and silicon are added to the vessel or ladle and aluminum to the ladle. Silicon combines with oxygen remaining after carbon deoxidation, and aluminum further deoxidizes the steel and combines with the nitrogen present. Manganese is added to meet specification requirements and to enhance forgeability. The phosphorus content of bessemer steel may be used to replace part of the carbon or manganese required to meet certain tensile properties. This practice results in a fully killed bessemer steel and has been described in more detail by Wright (**1**) Price (**2**), and Wilder (**3**).

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[3] Tentative Specifications for Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 - 48T), 1949 Book of A.S.T.M. Standards, Part 1, p. 39.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 780.

TABLE I.—CHEMICAL AND MECHANICAL PROPERTIES OF STEELS.

Melt	Process[a]	Deoxidation	Chemical Analysis, per cent					Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Austenitic Grain Size
			Carbon	Manganese	Phosphorus	Sulfur	Silicon				
A	O.H.	Silicon-aluminum	0.26	0.80	0.016	0.035	0.14	48 900	80 000	37	6 to 8
B	O.H.	Silicon-aluminum	0.24	0.83	0.018	0.033	0.14	47 100	77 800	41	6 to 8
C	O.H.	Silicon-aluminum	0.24	0.88	0.017	0.030	0.17	47 600	82 200	40	6 to 8
Average		Silicon-aluminum	0.25	0.84	0.017	...	...	47 900	80 000	39	6 to 8
D	O.H.	Silicon	0.25	0.80	0.018	0.033	0.16	46 200	78 500	39	2 to 4
E	O.H.	Silicon	0.28	0.87	0.018	0.033	0.19	46 800	82 700	39	2 to 4
F	O.H.	Silicon	0.28	0.71	0.018	0.028	0.18	45 300	79 000	40	2 to 4
Average		Silicon	0.27	0.79	0.018	...	...	46 100	80 100	39	2 to 4
G	B	Carbon-silicon-aluminum	0.16	0.51	0.075	0.022	0.22	45 000	71 900	41	7 to 8
H	B	Carbon-silicon-aluminum	0.15	0.44	0.077	0.023	0.22	47 000	72 500	42	7 to 8
I	B	Carbon-silicon-aluminum	0.14	0.43	0.075	0.020	0.22	46 000	73 500	38	7 to 8
J	B	Carbon-silicon-aluminum	0.13	0.43	0.075	0.023	0.21	45 600	71 200	43	7 to 8
K	B	Carbon-silicon-aluminum	0.14	0.42	0.073	0.022	0.21	45 700	71 400	43	7 to 8
L	B	Carbon-silicon-aluminum	0.13	0.44	0.076	0.020	0.19	43 600	71 000	43	7 to 8
Average		Carbon-silicon-aluminum	0.14	0.45	0.075	...	...	45 500	71 900	42	7 to 8
Minimum Requirements in Specification A 53 - 47								35 000	60 000	30	

[a] O.H. = Basic open-hearth.
B = Deoxidized acid-bessemer.

MATERIALS

All of the material used in this investigation was grade B seamless pipe $6\frac{5}{8}$ in. in outside diameter with a 0.562-in. wall made in accordance with A.S.T.M. Specifications A 53.[2] Various types of steel including 3 melts of silicon-killed basic open-hearth steel, 3 melts of silicon-aluminum-killed basic open-hearth steel, and 6 melts of deoxidized acid-bessemer steel were studied. All of these types of steel are used in the manufacture of seamless pipe and, therefore, a comparison of their properties should be of interest. The silicon killed open-hearth steel is of particular interest because of its improved creep and creep rupture properties (**4**). Open-hearth seamless pipe is generally manufactured from either silicon-aluminum-killed or aluminum-killed steels. The latter type of steel with reference to welding characteristics should, in general, be similar to silicon-aluminum-killed steels and, therefore, was not included in the investigation.

Chemical analysis, tensile properties and standard A.S.T.M. austenitic grain size[5] after carburizing the steels investigated are shown in Table I. The residual alloy content of these steels was very low as no alloy scrap was used in steel melting. Due to the higher phosphorus content of acid-bessemer steel, the carbon and manganese required to meet similar tensile properties is lower, compared with basic open-hearth steels. Grade B seamless pipe to A.S.T.M. Specification A 53[2] is usually manufactured so that the product is reasonably uniform with respect to tensile properties. All of the steels had a fine austenitic grain size with exception of the silicon-killed open-hearth steel which was coarse grained. All material was tested in the "as-rolled" condition. Hardenability tests of end-quenched 0.5 in. diameter solid bars made in accordance with A.S.T.M. Method A 255[6] are shown in Fig. 1. The hardenability curves represent the average results of all heats quenched from 1600 F. The bessemer steel had the lowest hardenability due to chemical composition and austenitic grain size. The silicon-killed open-hearth steels had the highest hardenability which may be associated with the chemical composition and coarse grain size. Hardenability results obtained with material quenched from 2100 F. were similar for the open-hearth steels. The bessemer steel, however, had a lower hardenability due to its chemical composition.

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[5] Standard Classifications of Austenite Grain Size in Steels (E 19 - 46), 1949 Book of A.S.T.M. Standards, Part 1, p. 1271.

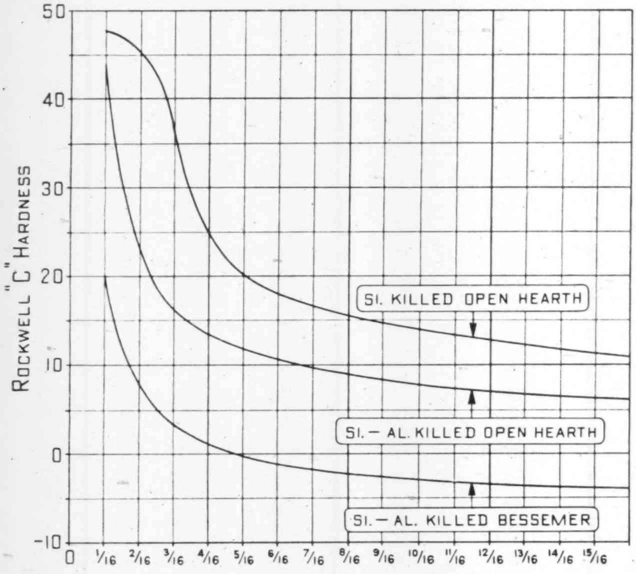

FIG. 1.—Hardenability of Grade B Steels.

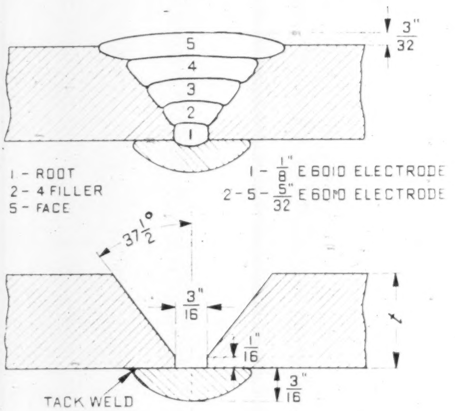

FIG. 2.—Welding Sequence.

TEST METHODS

Girth Welds, Tension, and Bend Tests:

Circumferential manual metallic arc welds of full pipe sections were used in this investigation. The welds were made with "as-rolled" pipe by a pipe fabricator in accordance with the A.S.M.E. Boiler Construction Code.[7] The type of joint and welding sequence are shown in Fig. 2. Preheat, postheat or stress relieving were not used during welding. The pipe was rolled during welding and welded in a flat position, test position 1 F in Fig. Q-27, Section IX of the A.S.M.E. Boiler Construction Code.

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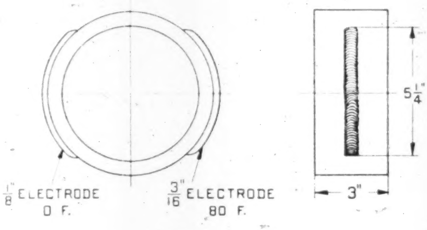
[6] Tentative Method of End-Quench Test for Hardenability of Steel (A 255 – 48T), 1949 Book of A.S.T.M. Standards, Part 1, p. 505.



TRANSVERSE BEAD

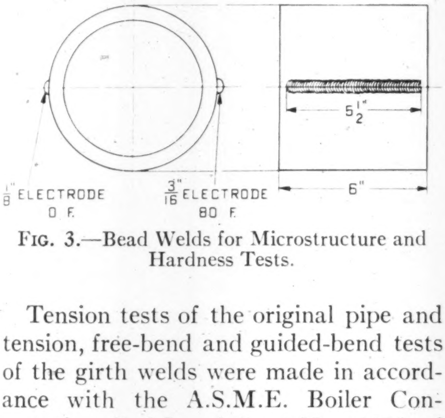
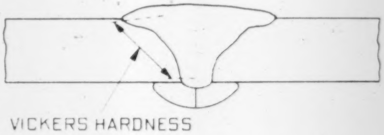
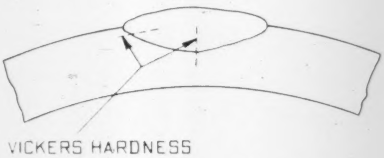


FIG. 3.—Bead Welds for Microstructure and Hardness Tests.

Tension tests of the original pipe and tension, free-bend and guided-bend tests of the girth welds were made in accordance with the A.S.M.E. Boiler Construction Code[7] and Case No. 1022 of the Code. Free-bend test specimens were 0.5 in. thick and 0.75 in. wide. The root- and face-guided bend test specimens were 0.5 in. thick and $1\frac{1}{2}$ in. wide. The guided-bend jig was modified in accordance with Case 1022. Free-bend and face-guided bend test specimens were not stress relieved, but after grinding they were aged at ambient temperature for one week. The grinding marks on the test specimens were longitudinal. The root-guided bend test specimens were stress relieved at 1200 F. and after grinding, the specimens were artificially aged at 240 F. for 20 hr. to relieve grinding stresses. Stress relieving of welded construction may be used by the pipe fabricator under certain conditions due to the wall and ductility characteristics of the material.



(GIRTH WELD)



(BEAD WELD)

FIG. 4.—Hardness Contours of Weld.

Bead Welds, Hardness Surveys, and Internal Structure:

In order to reduce variables associated with manual metallic arc welding, transverse and longitudinal single weld beads shown in Fig. 3 were made at the U. S. Steel Corp. Research Laboratory. These beads were deposited in a flat position with an automatic welding head with a stick electrode attachment. An E-6010 welding electrode was employed. The $\frac{3}{16}$-in. electrode was deposited at 80 F. with 180 amp. and 6 in. per min. travel speed. The $\frac{1}{8}$ in. electrode was deposited at 0 F. with 100 amp. and 10 in. per min. travel speed. The reason for depositing two beads under different welding conditions was to represent normal welding conditions with the $\frac{3}{16}$-in. electrode and severe welding condition with the $\frac{1}{8}$-in. electrode.

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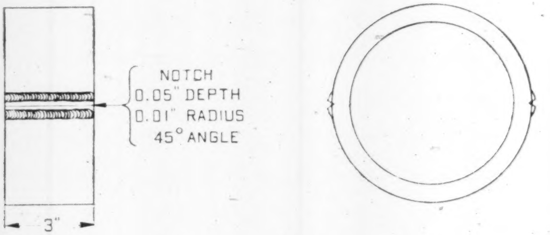
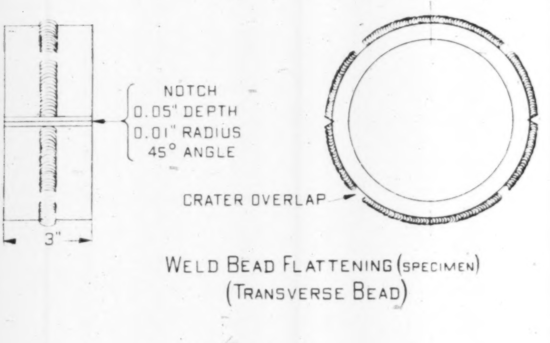
[7] Standard Qualification for Welding Procedure and Welding Operator, Section IX, Am. Soc. Mechanical Engrs., Boiler Construction Code (1949).

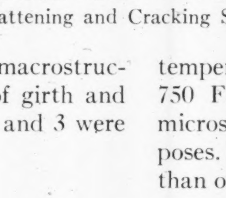
Hardness surveys were made on transverse sections of the girth and bead welds shown in Figs. 2 and 3. A standard Vickers hardness tester was used with 1- and 10-kg. loads. The 1-kg. load was used only in the heat affected zone and the hardness contours are shown in Fig. 4. The microstructure and macrostructure of transverse sections of girth and bead welds shown in Figs. 2 and 3 were examined.



WELD BEAD FLATTENING (SPECIMEN)
(LONGITUDINAL BEAD)



WELD BEAD FLATTENING (SPECIMEN)
(TRANSVERSE BEAD)



CRACKING SUSCEPTIBILITY TEST SPECIMEN

FIG. 5.—Weld Bead Flattening and Cracking Susceptibility Test Specimens.

Impact Tests:

Standard Charpy keyhole impact tests were made in accordance with the A.S.M.E. Unfired Pressure Vessels Code.[8] Tests were made with a notch in the unaffected base material, with a notch in the heat-affected zone of the girth welds, and with a notch in the weld metal of the girth welds. Impact tests from each melt of steel were made at temperatures of −20, 0, 25, 75, 450, and 750 F. Material with a representative microstructure was selected for test purposes. At several temperatures more than one impact test was made, and this was done to be certain of the temperature-impact relationships reported.

---

[8] Rules for Construction of Unfired Pressure Vessels, Section VIII, Am. Soc. Mechanical Engrs., Boiler Construction Code (1950).

Cracking Susceptibility:

A weld bead shown in Fig. 5 was deposited in a flat position at 0 F. using a $\frac{1}{8}$-in. E-6010 electrode with 100 amp. and 10 in. per min. travel speed with the automatic welding head previously described. After welding, the specimen was heated at 1100 F. for 1 hr. and then sectioned longitudinally through the weld. The vertical plane through the center of the weld was ground and polished, etched with ammonium persulfate to locate the heat-affected zone of the weld. The etched specimen was magnaflux tested and the lengths of cracks were measured under the microscope.

The results were expressed in percentage of total weld length cracked. A bead $1\frac{1}{4}$ in. long was employed so that the conditions encountered in striking an arc in the absence of preheat were obtained. Weld beads with a greater length were not employed because they are appreciably preheated by the weld deposit and the susceptibility to cracking is decreased.

Weld Bead Flattening Tests:

Weld bead flattening tests were made from pipe rings shown in Fig. 5. Weld beads were deposited in a flat position on pipe rings 6 in. wide with the automatic welding head previously described. A $\frac{3}{16}$-in. E-6010 electrode with 180 amp. and 6 in. per min. travel speed was used. The temperature at the start of each weld bead was 80 F. The weld beads were deposited in the longitudinal direction with respect to the major axis of the pipe. After welding, the beads were notched as shown in Fig. 5 in accordance with the procedure outlined by Offenhauer and Koopman (5) and aged at 212 F. for 24 hr. The notched pipe rings with the notch at 90 deg. from the direction of applied force were flattened by compression in a tension testing machine at the rate of 0.5 in. per min. until a crack appeared. The distance between plates used in flattening, lateral contraction in width of the specimen and the maximum load were measured to provide a method for evaluating weldability of a pipe section. The use of an automatic welding head eliminated certain variables encountered in girth welding. The base of the notch was in weld metal, and, under these conditions, the crack originating in the notch penetrated into the heat-affected zone of the base material before failure was recorded. The notch, therefore, in the heat-affected zone before failure of the specimen consisted of a crack which originated in the weld metal. Pipe rings without a notch in the unwelded and welded condition were also tested.

RESULTS AND DISCUSSION

Tension and Bend Test Results of Girth Welds:

Tensile properties of the pipe material in the "as-rolled" condition are shown in Table I. All of the material met A.S.T.M. requirements for pipe.[2] It will be observed that the yield strength and percentage elongation of all the steels were similar. Tensile strengths of the open-hearth steels were higher than the bessemer steel, although all the steels were well above the specification requirements.

Pipe used by fabricators is generally welded and frequently must meet the requirements of the A.S.M.E. Boiler Construction Code. In order to determine if the material used in this investigation would meet these requirements, girth welds were tested. Tension tests of girth welds made in accordance with the welding procedure outlined in Fig. 2 are shown in Table II. All of the steels

TABLE II.—TENSION AND BEND TEST RESULTS OF GIRTH WELDS.

Material	Tension Tests			Bend Tests		
	Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Free-Bend Test elongation, percent	Guided-Bend	
					Face	Root^a
Melts A to C, incl. (silicon-aluminum-killed open-hearth)	49 800	74 000	20	73	OK	OK
Melts D to F, incl. (silicon killed open-hearth)	48 900	74 500	17	67	OK	OK
Melts G to L, incl. (deoxidized bessemer)	49 100	72 900	25	57	OK	OK
A.S.M.E. Boiler Code minimum requirements	...	60 000	...	25		...

^{a} Stress-relieved at 1200 F.

TABLE III.—GIRTH AND BEAD WELD VICKERS HARDNESS.

	Girth Weld			Normal Welding Procedure, Longitudinal Bead Weld			Severe Welding Procedure, Longitudinal Bead Weld		
	1^a	2^a	3^a	1^a	2^a	3^a	1^a	2^a	3^a
MELTS A TO C, INCL., SILICON-ALUMINUM-KILLED OPEN-HEARTH									
Range	146 to 171	145 to 219	134 to 170	155 to 180	224 to 276	179 to 198	159 to 175	330 to 401	248 to 285
Average	158	172	150	170	252	189	164	363	272
MELTS D TO F, INCL., SILICON-KILLED OPEN-HEARTH									
Range	140 to 181	142 to 219	132 to 168	172 to 186	214 to 283	187 to 215	160 to 183	283 to 339	251 to 281
Average	164	180	147	178	253	197	171	309	263
MELTS G TO L, INCL., DEOXIDIZED ACID-BESSEMER									
Range	138 to 168	143 to 197	133 to 167	148 to 178	193 to 239	191 to 225	147 to 165	210 to 263	262 to 287
Average	152	171	148	160	221	204	157	234	271

^{a} 1 = Unaffected base metal.
2 = Heat-affected zone.
3 = Weld metal.

met requirements of the Boiler Code. The percentage elongation was lower for the open-hearth steels, particularly the silicon-killed steel.

Free-bend tests of all the steels were well within Boiler Code requirements. Lower total elongation was observed in the bessemer steel. In the guided-bend jig all the face-bend tests were satisfactory. Some of the open-hearth and bessemer steels in the "as-welded" condition failed in the root-bend test. After stress relieving at 1200 F., the root-bend specimens were all satisfactory. A metallurgical examination of the girthwelds indicated good penetration and minimum porosity in the root beads. The heavy wall thickness was associated with the necessity for stress relieving the root-bend test specimens.

Free-bend tests are used to evaluate ductility of welds, but it should be pointed out that the tension test may also be employed to evaluate this property. In the tension test the entire cross-section of the weld is uniformly stressed while in the free-bend test the outer fibers of the test specimen are subjected to maximum elongation.

In the root- and face-guided bend tests, soundness is evaluated in the outer fibers of the test specimen, and the internal structure of the weld is not observed unless the specimen is fractured. The side-bend test of a well evaluates soundness throughout the welded area, but is employed only in material with a $\frac{3}{4}$-in. or heavier wall. With reference to the soundness of a weld, the nick-break test in lighter wall material offers certain possibilities which have not been generally recognized. Although nick-break tests were not used in this investigation, macrostructure and microstructure of the welds were examined.

Methods for evaluating the ductility and soundness of welded structure should be re-evaluated, particularly in the higher strength materials. Further, the notch effects obtained in the root bead with or without a back-up ring and in the face bead at the junction of the weld metal and base material are significant factors in considering strength of the welded joint.

Hardness Results of Girth and Bead Welds:

Vickers hardness results of the girth welds, as given in Table III, were similar. The maximum hardness was observed in the heat-affected zone of the base metal adjacent to the face bead. The hardness contours are shown in Fig. 4. Unless a small hardness indentation is used in the heat-affected zone, the maximum hardness may not be observed. The weld metal of the face bead had a greater hardness compared with the root and filler weld beads. Hardness of the heat-affected zone adjacent to the root and filler beads was lowered by heat input from deposition of the several weld beads.

Due to the welding sequence shown in Fig. 2, hardness of the heat-affected area associated with the face bead was the only significant area in the weld where hardness might be associated with adverse weld performance. It should be pointed out, however, that in depositing the root bead the hardness originally obtained may, in certain steels, be related to cracking unless preheat is employed.

Hardness results of the longitudinal weld beads (Fig. 3) with normal and severe welding procedures are also given in Table III. Hardness contours are depicted in Fig. 4. The hardness results obtained with the longitudinal and transverse weld beads were similar, and therefore only the longitudinal weld bead results are discussed.

It will be observed in Table III that with normal welding procedure the maximum hardness of the open-hearth steels was greater compared with the bessemer steel. The maximum hardness of the welds was observed in the heat-affected area adjacent to the weld bead and at the base of the bead

With the severe welding procedure, the maximum hardness in the bead welds was again observed in the open-hearth steels and particularly in the silicon-aluminum-killed steel. Hardness of the weld metal was appreciably higher in weld beads with the severe welding procedure. The lower hardness observed in the bessemer steel may be associated with the lower carbon content (Table I) and lower hardenability (Fig. 1) of this type of steel.

Microstructure of Girth and Bead Welds:

Girth welds were metallographically examined, and typical structures are shown in Figs. 6 and 7. The silicon-aluminum-killed open-hearth steel in Fig. 6 was similar to the silicon-killed open-hearth steel. In comparing the bessemer steel in Fig. 7 with Fig. 6, it will be observed that the principal difference in structure may be associated with lower carbon content of the bessemer steel.

Weld metal in the root and filler beads was recrystallized, and the face bead was the only weld bead with an "as-cast" structure. The microstructure of the heat-affected zone was closely related to the hardness results obtained. The heat-affected zone of the base metal associated with the face bead was the only area of the base metal adjacent to the girth weld where the coarse-grained structure was observed. The metallographic structure depicted clearly indi-

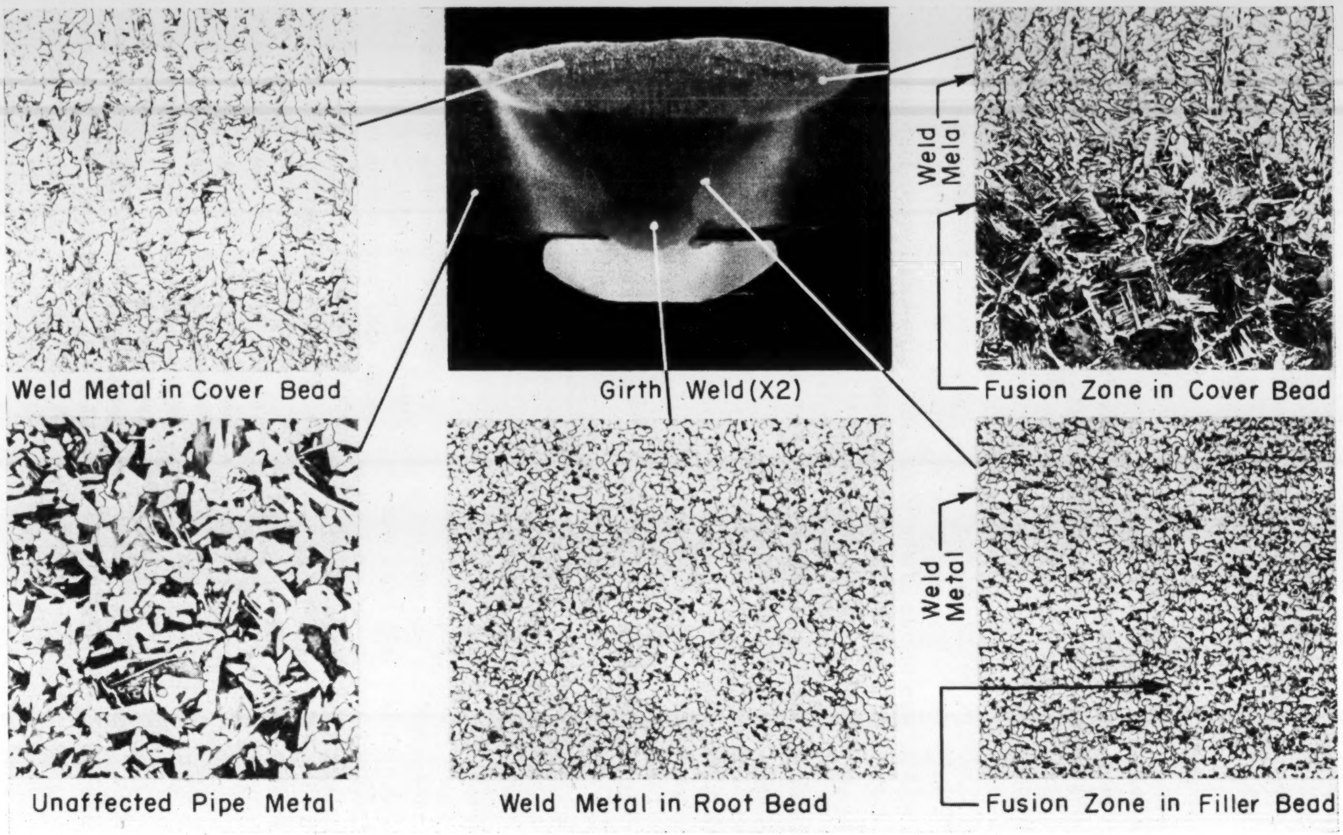

Weld Metal in Cover Bead
Girth Weld (X2)
Weld Metal
Fusion Zone in Cover Bead
Unaffected Pipe Metal
Weld Metal in Root Bead
Weld Metal
Fusion Zone in Filler Bead

FIG. 6.—Microstructure of Girth Weld Silicon-Aluminum-Killed Open-Hearth Steel (×100). Etched in Nital.

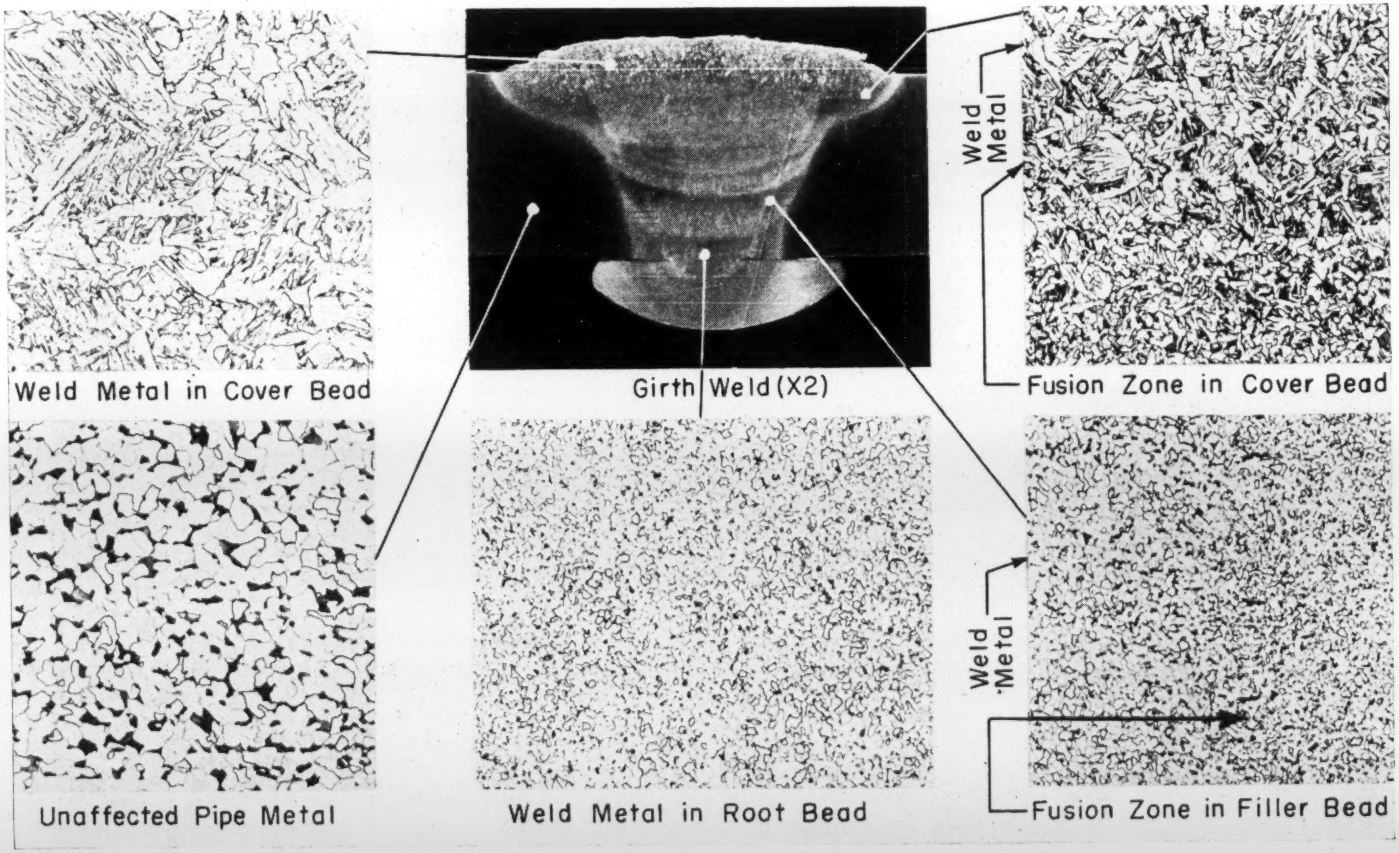

Weld Metal in Cover Bead
Girth Weld (X2)
Weld Metal
Fusion Zone in Cover Bead
Unaffected Pipe Metal
Weld Metal in Root Bead
Weld Metal
Fusion Zone in Filler Bead

Fig. 7.—Microstructure of Girth Weld, Deoxidized Acid-Bessemer Steel (×100). Etched in Nital.

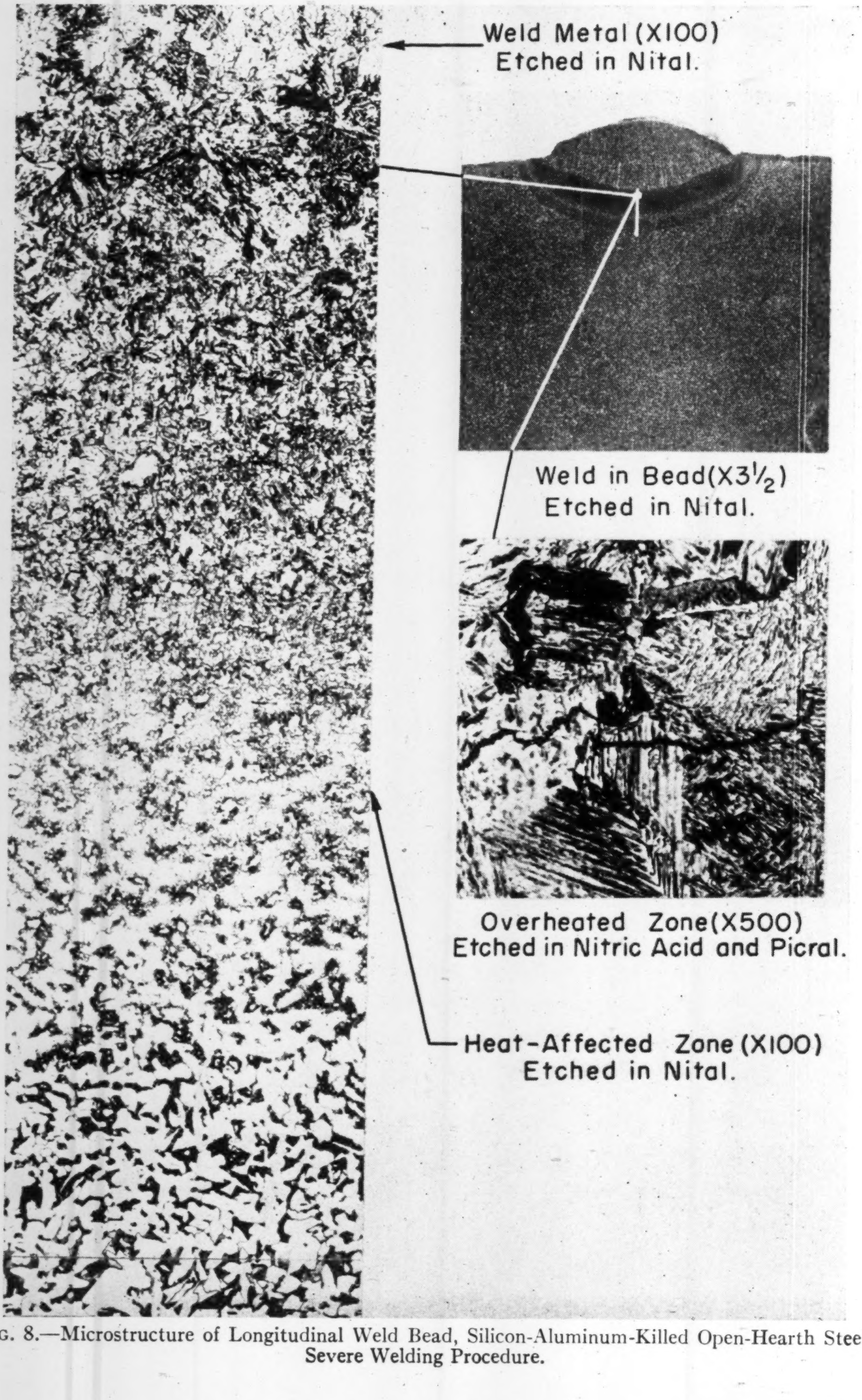

Weld Metal (X100)
Etched in Nital.
Weld in Bead (X3½)
Etched in Nital.
Overheated Zone (X500)
Etched in Nitric Acid and Picral.
Heat-Affected Zone (X100)
Etched in Nital.

FIG. 8.—Microstructure of Longitudinal Weld Bead, Silicon-Aluminum-Killed Open-Hearth Steel. Severe Welding Procedure.

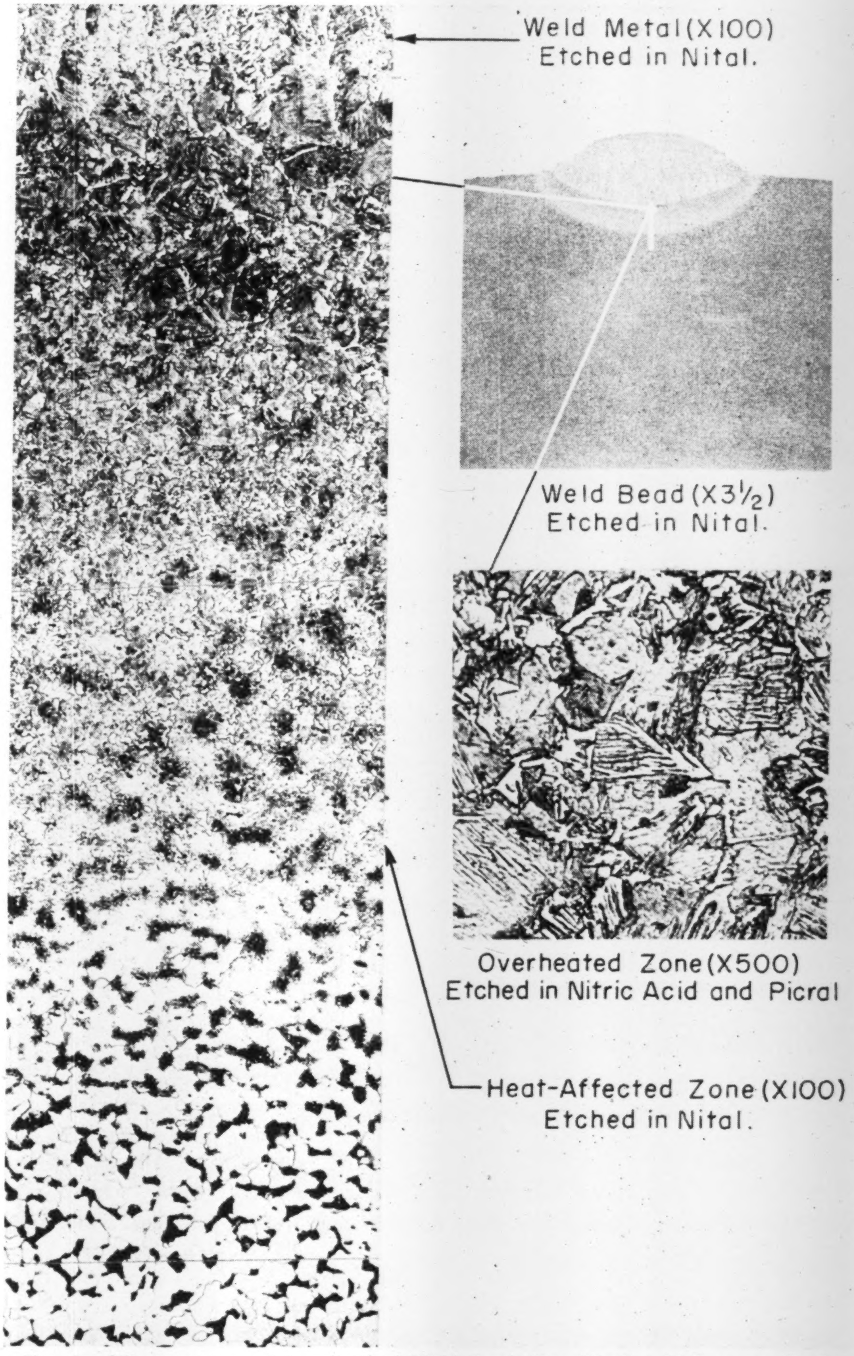
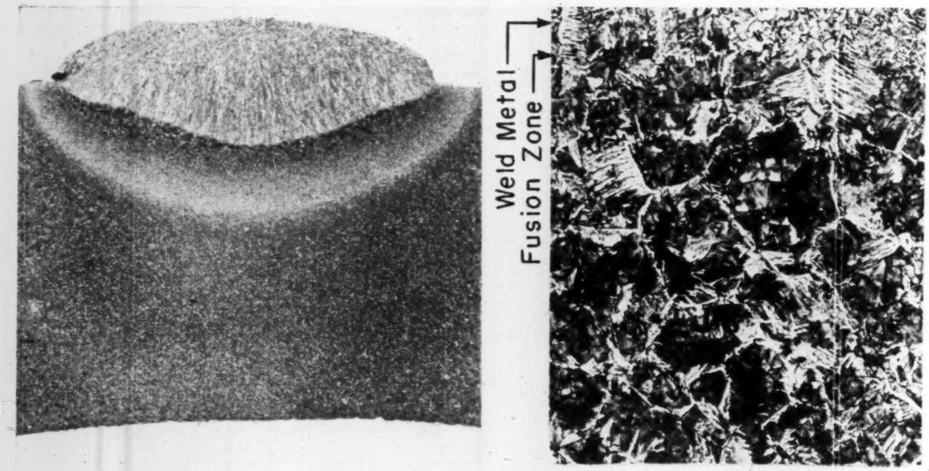
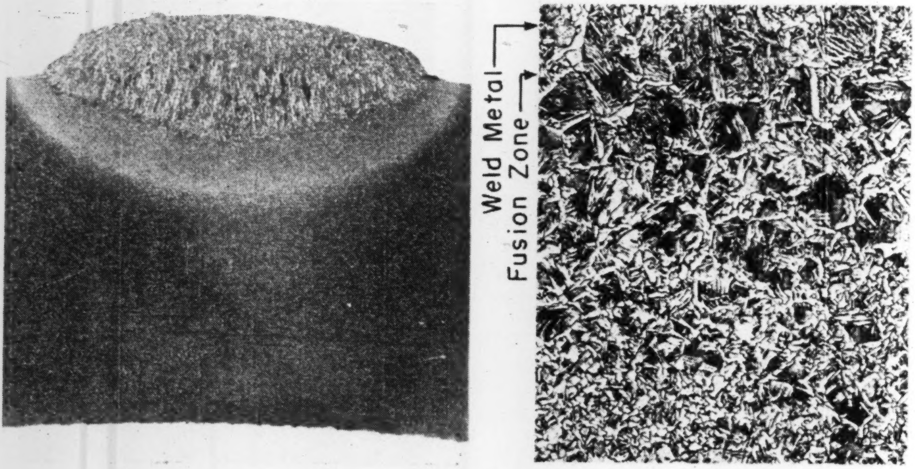

Weld Metal (X100)
Etched in Nital.
Weld Bead (X3½)
Etched in Nital.
Overheated Zone (X500)
Etched in Nitric Acid and Picral
Heat-Affected Zone (X100)
Etched in Nital.

FIG. 9.—Microstructure of Longitudinal Weld Bead, Deoxidized Acid-Bessemer Steel. Severe Welding Procedure.


Weld Metal
Fusion Zone

Silicon–Aluminum Killed Open-Hearth Steel

Weld Bead(X3½) Fusion Zone(X100)


Weld Metal
Fusion Zone

Deoxidized Acid-Bessemer Steel

Weld Bead (X3½) Fusion Zone (X100)

FIG. 10.—Microstructure of Longitudinal Weld Beads. Normal Welding Procedure. Etched in Nital

cates the refinement of structure obtained in multi-pass welding.

Typical microstructures observed in longitudinal weld beads of the silicon-aluminum-killed open-hearth and bessemer steels with severe welding procedure are shown in Figs. 8 and 9. Microstructure of the silicon-killed open-hearth steel was similar to the silicon-aluminum-killed open-hearth steel. Circumferential weld bead results are not shown as they were comparable with the longitudinal weld beads.

Acicular martensite and bainite were observed in the heat-affected zone of the base metal near the weld metal in all weld beads deposited with a severe welding procedure. The open-hearth steels were completely acicular in the base metal near the fusion zone, but in the bessemer steel pearlite and free ferrite were also associated with the acicular structure.

Underbead cracks were observed in the open-hearth steel and are shown in Fig. 8. No underbead cracks were observed in the bessemer steel. Weld metal in all weld beads had a cast columnar structure. In the heat-affected zone of the base metal, various structures associated with the thermal gradient were observed. Adjacent to the area of grain growth, a completely recrystallized structure with grain refinement was observed. Partial recrystallization and spheroidized structures merging into unaffected base material were observed.

The metallographic structure of weld beads with normal welding procedure is shown in Fig. 10. The silicon-killed open-hearth steel was similar to the silicon-aluminum-killed steel shown in Fig. 10. Circumferential weld beads in all the specimens were similar to the longitudinal weld beads depicted in Fig. 10. The heat-affected zones of the large weld beads were appreciably larger compared with the heat-affected zones of the small weld beads shown in the macrographs of Figs. 8 and 9. The large heat-affected zone is associated with the greater heat input and slower cooling rate. Martensite and underbead cracking were therefore not observed in the weld beads with normal welding procedure.

A columnar cast structure may be observed in the weld metal of Fig. 10. Immediately beneath the weld metal, coarse grains with free ferrite and a Widmanstätten structure associated with the grain boundaries were observed. The coarse grain structure gradually decreased in size, merging into an area of recrystallized metal with a fine grain structure. The fine grain structure merged into a partially recrystallized structure with some spheroidization and finally the unaffected base material. The metallographic structure of the bessemer steel indicated a smaller area of coarse-grained structure compared with the open-hearth steels and less pearlite was present due to the lower carbon content of the bessemer steel.

Impact Properties of Girth Welds:

The steels in this investigation were studied in the "as-rolled" state although they are not used in this condition for low-temperature service. In the A.S.M.E. Boiler Construction Code, impact tests are required only for operating temperatures below −20 F. The production of steel for low-temperature service requires special deoxidation practice in conjunction with heat treatment and frequently necessitates the use of alloy additions. The impact characteristics of various types of steel have been discussed, by one of the authors (6), and in the present paper the impact properties of girth welds at −20 F. to 750 F. of steels ordinarily used for these operating temperatures are reported.

Although the steels in this investigation are frequently employed at temperatures above 750 F., the creep properties (7) and graphitization characteristics (8) are of such a nature that chromium molybdenum steels are generally used for higher operating temperatures. Also, it is recognized that in studying the impact properties of girth welds, some uncertainty exists in determining the impact properties of the heat-affected zone. In the discussion of metallographic structure of the welds in this investigation, it was observed that the coarse-grained structure in the heat-affected zone was only in the area adjacent to the face or cover bead. The remainder of the heat-affected zone in the weld had a fine grained structure. Due to the fact that grain size has an important bearing on impact properties, the girth welds in this investigation were notched in the heat-affected zone so that the path of fracture would include the coarse-grained structure. This is illustrated in Fig. 11.

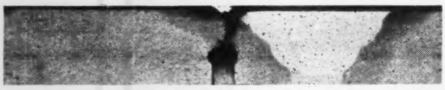


FIG. 11.—Location of Fracture in Impact Specimen of Heat-Affected Zone of Girth Welds.

TABLE IV.—AVERAGE CHARPY IMPACT TESTS OF GIRTH WELDS.

Testing Temperature, deg. Fahr.	Specimen (Standard Size), ft. lb.								
	Unaffected Base Metal			Heat-Affected Zone			Weld Metal		
	1^a	2^a	3^a	1^a	2^a	3^a	1^a	2^a	3^a
−20	20	5	23	47	32	36	29	29	30
0	22	6	24	49	38	38	31	30	33
25	26	23	28	48	40	40	34	32	37
75	32	28	32	52	43	48	33	35	38
450	31	32	43	43	44	45	32	32	34
750	18	19	25	26	23	27	18	16	17

[a] 1 = Melts A to C, incl., silicon-aluminum-killed open-hearth, average of three impact tests.
2 = Melts D to F, incl., silicon-killed open-hearth, average of three impact tests.
3 = Melts G to L, incl., deoxidized acid-bessemer, average of six impact tests.

The weld metal impact specimens were notched from the root bead side of the specimen, and fracture occurred entirely in weld metal. Location of the notch in impact specimens of "as-rolled" pipe is illustrated in Fig. 1 of the earlier paper by Wilder (6).

The impact properties are summarized in Table IV. It will be observed that in the weld metal and heat-affected zone the impact properties for all steels were similar. At 750 F., lower impact properties were observed in these zones although the specimens were not brittle at this temperature.

TABLE V.—WELD BEAD FLATTENING TEST RESULTS.

Type[1]	Contraction, per cent	Max. Load, 1000 lb.	Flattening, percentage outside diameter[2]
MELTS A TO C, INCL., SILICON-ALUMINUM-KILLED OPEN-HEARTH			
U	3.2	35.2	48
U-CW	2.4	37.5	54
U-LW	1.5	35.9	51
N-LW	1.5	32.4	52
N	0.8	18.7	84
N-CW	0.5	19.8	88
MELTS D TO F, INCL., SILICON-KILLED OPEN-HEARTH			
U	2.6	30.0	56
U-CW	2.2	33.5	56
U-LW	1.3	32.5	56
N-LW	1.6	33.2	54
N	0.8	19.4	82
N-CW	0.5	19.4	88
MELTS G TO L, INCL., DEOXIDIZED ACID-BESSEMER			
U	3.3	35.5	49
U-CW	3.3	42.4	50
U-LW	1.5	38.2	48
N-LW	1.4	30.8	57
N	1.0	19.6	80
N-CW	0.4	19.5	89

[1] U = Unnotched,
N = Notched,
CW = Circumferential weld bead, and
LW = Longitudinal weld bead.

[2] Percentage outside diameter =

$$\frac{\text{distance between plates, in.} \times 100}{6.625}.$$

In the original pipe material, the silicon-killed open-hearth steel was brittle at 0 F. and at −20 F., while the silicon-aluminum-killed open-hearth and bessemer steels had satisfactory impact properties. These results indicate that in the heat-affected zone of girth welds in lighter wall pipe, the properties of silicon-killed open-hearth steel at low temperature may be lower than those shown in Table IV. At 750 F. the impact properties were lower for all the steels as compared with 450 F.

Weld Bead Flattening Test Results:

The flattening test specimens shown in Fig. 5 were used in an attempt to evaluate weldability with particular reference to tubular products. A flattening test specimen consisting of a ring or nipple of pipe is a recognized means of measuring the ductility of commercial pipe and tubing.

The results shown in Table V indicate that weldability as evaluated by this type of test was similar for all the steels investigated. The longitudinal weld bead in the notched condition was the most satisfactory type of test due to the fact that a rapid rate of crack propagation occurred at the time of fracture. The notched longitudinal weld bead results indicated that at ambient temperature the ductility of the various materials as measured by percentage flattening and percentage contraction was similar. Results obtained with the other types of specimens were erratic due to the formation of hairline cracks which were difficult to detect at the time of their initial formation.

A comparison of the notched longitudinal weld bead tests with unnotched, unwelded specimens indicates that the amount of flattening was similar but the percentage contraction was less in the welded specimens. A more complete evaluation of weldability would be obtained if the tests were conducted at various temperatures. Under this condition, a transition temperature as described by Offenhauer and Koopman (5) would be determined and weldability of the steels could be more accurately evaluated. However, with reference to future testing, results of this investigation indicate that only the notched longitudinal weld bead specimens should be employed in evaluating weldability.

Cracking Susceptibility Test Results:

The tendency for underbead cracking in weld bead specimens was evaluated with the specimen shown in Fig. 5. The results are reported in Table VI. The type of crack observed in the bead welds is shown in Fig. 8.

Underbead cracks were observed in the open-hearth steels and the silicon-killed steel had the greatest cracking susceptibility. No cracks were observed in the bessemer steel. These results are related to the hardenability and hardness results previously discussed. The bessemer steel had the lowest hardenability when quenched from 1600 F. and 2100 F. and in the weld beads deposited with severe welding procedure the lowest hardness. The bessemer steel, due to its higher phosphorus content, also had a lower carbon and manganese content which apparently is related to the lower cracking susceptibility. If a lime-coated low-hydrogen weld rod had been employed, it is quite possible that underbead cracks would not have been observed in the open-hearth steels.

TABLE VI.—CRACKING SUSCEPTIBILITY TEST RESULTS.

Materials	Percentage Cracked
Melts A to C, incl. (silicon-aluminum-killed open-hearth)	32
Melts D to F, incl. (silicon-killed open-hearth)	51
Melts G to L, incl. (deoxidized bessemer)	0

Conclusions

1. All materials tested were seamless pipes in the "as-rolled" condition and the chemical and physical properties met A.S.T.M. specification requirements for grade B steel pipe.[2] The bessemer seamless pipe was made from a special deoxidized steel, and although the carbon and manganese content were lower

compared with the open-hearth steels, the tensile properties were similar.

2. The deoxidized bessemer steel had the lowest hardenability when quenched from 1600 F. and 2100 F.

3. The silicon-aluminum-killed open-hearth and deoxidized bessemer steels had a fine austenitic grain size after carburizing at 1700 F. and the silicon-killed open-hearth steel was coarse grained.

4. Girth welds met the tensile and bend test requirements of the A.S.M.E. Boiler Construction Code.

5. Maximum hardness results in the girth and bead welds were higher in the open-hearth steels compared with the deoxidized bessemer steel.

6. Hardness and microstructure of longitudinal and circumferential weld beads were similar.

7. A coarse-grained structure was observed in the heat-affected zone of the bead welds and also adjoining the face beads of the girth welds in contrast to a fine-grained structure adjoining the filler and root beads of the girth welds.

8. Satisfactory impact properties in the girth welds were obtained at −20 F. to 750 F. in all the steels except the silicon-killed open-hearth steel at the lower temperatures.

9. In the weld bead flattening tests, all the steels were similar and the notched longitudinal weld bead tests were the most consistent.

10. The open-hearth steels exhibited underbead cracking in the cracking-susceptibility tests. Cracks were not observed in the bessemer steel.

Acknowledgments:

The authors acknowledge the encouragement received from E. G. Price, Vice-President in Charge of Operations, and the assistance and cooperation of both the United States Steel Corp. of Delaware and National Tube Co. Research Labs., and the operating personnel of National Tube Co., in conducting the investigation. They are particularly indebted to W. A. Barres of National Works, under whose direction the experimental work was conducted.

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1

DISCUSSION

Messrs. Perry R. Cassidy,[1] O. R. Carpenter,[2] and F. Eberle[3] (*presented in written form*).—The authors have presented data which in our opinion demonstrate that deoxidized acid-bessemer pipe of the quality stated in the paper can be satisfactorily welded into piping systems and for certain other pressure parts. It is our understanding that such use would be limited to 750 F.

We should like to know the authors' opinion as to the reproducibility of homogeneous quality of this type of steel in different mills and possibly different companies that might be licensed to produce the pipe.

The spread in tensile strength of 7000 to 10,000 psi. between the bessemer and open-hearth pipe seems unfortunate. If the open-hearth pipe selected for test had been near the tensility of the bessemer, and with fine grain size, the hardenability and the underbead cracking tendency might have been nearer in comparison. A.S.T.M. Specifications A 53, grade B pipe should in general average somewhat under 70,000 psi. tensile strength, and relatively few heats should reach 80,000 psi. Notwithstanding this difference in tensility, the elongation of the open-hearth pipe was equal to the bessemer.

Is it the authors' opinion that the bessemer pipe as welded has no materially different quench age-hardening characteristics than the open-hearth pipe?

Have the authors any data on the fatigue characteristics of welded bessemer as compared to welded open-hearth pipe, or for the base material without welding?

In introducing a new type of material such as the deoxidized acid-bessemer pipe to be used interchangeably with older types, it is well to proceed with caution until experience and laboratory tests such as described by the authors indicate what is safe and what may be questionable. The authors are to be congratulated upon their continuation of the exploration of the characteristics of this new type of steel pipe.

Messrs. A. B. Wilder, W. B. Kennedy, and F. W. Crouch (*authors*).—We offer the following comments regarding the significant points raised by Messrs. Cassidy, Carpenter and Eberle:

Where graphitization is a problem, we do not recommend the use of either open-hearth or bessemer plain carbon steels above an operating temperature of 750 F. Steels containing chromium should be used when graphitization is a problem. Although plain carbon steels, under certain conditions, are used at temperatures exceeding 1100 F., where creep is a problem at elevated temperatures, steels containing molybdenum should be employed and if oxidation is also a factor, chromium should be added.

With reference to the quality of bessemer seamless pipe, we have manufactured over a million tons of this type of material in our steel making plants, and have consistently met standard specification requirements for seamless pipe. Several other steel makers have manufactured killed bessemer seamless pipe and, as far as we know, quality of the product has been similar to our own product. In order to manufacture consistently on a sound economic basis either open-hearth or bessemer steel for seamless pipe, it is necessary to exercise many precautions. Our company has been a pioneer in the use of statistical quality control methods in steel making, and these principles have been adapted to the control of steel making practices. In addition, experience over a period of many years in the manufacture of the seamless product has been very helpful in developing a satisfactory bessemer steel making practice.

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[2] Director, Works Control Lab., The Babcock & Wilcox Co., Barberton, Ohio.

[3] Chief Metallurgist, Research and Development Dept., The Babcock & Wilcox Co., New York, N. Y.

The ratio of the yield strength to the tensile strength is higher for bessemer seamless compared with open-hearth seamless pipe. In melting open-hearth steel for seamless pipe with a 0.562 in. wall, yield strength is the controlling factor. It will be observed that the yield strengths and elongations of the open-hearth and bessemer heats were similar. If open-hearth steel were melted to a lower chemistry, certain melts would fail to meet the minimum yield strength requirements of 35,000 psi. Messrs. Cassidy, Carpenter, and Eberle state that the average tensile strength of Specifications A 53 grade B material is about 70,000 psi., and this would be true for lighter wall open-hearth material with lower carbon or manganese. We feel the comparison of underhead cracking is valid, as the material studied in this investigation is representative of what would be normally manufactured.

No data are available on the quench age-hardening characteristics or fatigue properties of the material investigated. It is planned to obtain this information. A limited number of fatigue tests conducted several years ago indicate that the fatigue characteristics of open-hearth and bessemer seamless pipe are similar.

MESSRS. C. F. HULINGS AND JOHN HOWELL[4] (*presented in written form*).—This paper on the "Welding Characteristics of 'Open' Hearth and Bessemer Steel Pipe" portrays another victory for the survival of bessemer in the steelmaking field. Obsolescence of this pioneer steelmaking technique has been threatened several times, due to improvements in open-hearth practice made necessary to meet consumers demands for better ductility, formability, and other steel qualities in this category.

Welding, however, has always been a quality for which bessemer steel has often been specified in preference to open-hearth. It might be mentioned also in passing that bessemer steel never suffered by comparison where machinability was the primary quality demanded for a specific part. It is not surprising then that this improved deoxidized bessemer steel product has met the welding requirements described by Messrs. Wilder, Kennedy, and Crouch.

End use of steel for wartime products made manufacturers and consumers more practice conscious. Many steel users at that time first heard of the various steel-making practices such as rimmed, capped, semi-killed, fully killed or, as described here, fully deoxidized. Wilder in 1948 did another excellent job of preparing a paper entitled "Deoxidized Acid Bessemer Grade B Seamless Pipe—Standard Specifications and Codes" describing comparative steps in the manufacture of regular and deoxidized bessemer steel. He analyzed the favorable results of both laboratory tests and fabrication operations designed to compare the quality of this special bessemer steel with the usually more favored open-hearth steel.

The secret of the satisfactory performance has been proved to be thorough deoxidation. This does not necessarily mean, however, that any one particular method of deoxidation is mandatory for satisfactory results. Although our laboratory tests were not so extensive as those described by the present investigators, we completed a program of weld, bend, Van Stone lap, and swage tests on bessemer steel thoroughly deoxidized but by a method different from that reported by Wilder and associates.

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[4] Metallurgical Dept., Jones & Laughlin Steel Corp., Aliquippa, Pa.

In this program, samples of $8\frac{5}{8}$ in. grade B seamless pipe from ten blows of deoxidized bessemer steel were compared with four grade B open-hearth heats with very favorable results.

Bessemer pipe was welded to bessemer, open-hearth to open-hearth and bessemer to open-hearth to cover all possible practical commercial combinations. Etch, microstructure, Rockwell and tension tests were made on all combinations. Results of these tests may be briefly summarized as follows:

(*a*) Full-section weld tests were etched in acid and no defective welds were found.

(*b*) Microexamination of welds showed good fusion between weld and pipe metal with no abnormal heat penetration.

(*c*) Rockwell exploration on strip tests showed very little hardening in the heat-affected zone on any of the tests, indicating that the deoxidized bessemer had the same degree of insensitivity to hardening during welding as grade B open-hearth steel.

(*d*) Longitudinal tension test coupons, with weld metal intact, were tested to failure with weld transverse to load. All failures occurred outside the weld and also outside the heat-affected area in every case. Tension test values of the bessemer pipe were equal or superior to those found on the open-hearth samples.

Another weld test included the fabrication of two boiler headers made up of samples from eight deoxidized bessemer blows. Each boiler header consisted of a main header tube with three necks welded thereto by four weld passes. Warping and pulling out of a vertical plane occurs during welding, and after cooling all necks were straightened back to their original position. This is practically a reverse bend, and this severe test was successfully met by all samples without cracks, tears, weld rupture, or failure of any nature.

In addition to these weld tests, deoxidized bessemer steel was found equal to open-hearth in hot and cold bending, Van Stone laps and hot swaging ($8\frac{5}{8}$ to $4\frac{1}{2}$ in. outside diameter) operations.

Our results, therefore, confirm those described by Wilder and associates and were so favorable that this special bessemer steel is now approved for application on seamless orders specifying grade B on API Specifications 5 L or A.S.T.M. Specifications A 53 and grade H casing on API Specifications 5 A. As stated above, complete deoxidation is absolutely necessary, and it is our practice to definitely confirm this by requiring fine grain results of McQuaid-Ehn tests on all blows applied on seamless pipe.

MESSRS. WILDER, KENNEDY, AND CROUCH (*authors*).— The remarks by Messrs. John Howell and C. F. Hulings are of particular interest because they summarize some of the results obtained on grade B bessemer seamless pipe made by another major steel producer, Jones & Laughlin Steel Corp. Although the deoxidation practice differed from that described in our paper, the fact that a fine-grained steel was produced is the most important factor with respect to the properties of the material.

With reference to this matter of deoxidation, the steel maker must develop a practice which not only permits the economic production of seamless pipe within the producing unit, but properties of the material must also be satisfactory for the end use. The two problems are essentially dependent upon chemical composition and deoxidation practice and not necessarily the steel-making process. The practice developed for one plant may not necessarily produce a satisfactory product in another plant. For example, certain grades of seamless pipe are produced on Pilger mills in Europe from rimmed steel. We have not been able consistently to produce on an economic basis seamless pipe from rimmed steel by the Manassmen process. Further, if we were able to produce seamless pipe from rimmed steel, it would be necessary, in the case of bessemer steel, to modify the practice in order to achieve the beneficial results obtained from fine-grained deoxidation practice.

Messrs. Howell and Hulings refer to the fabricating characteristics of bessemer seamless and state that the material may be satisfactorily welded, bent, Van Stoned and swaged. We have also observed the fabrication of bessemer seamless pipe in many fabricating shops throughout the country and have found the material to be satisfactory. Further, a large amount of bessemer seamless pipe has been successfully used in the construction of gas and oil transmission lines and for casing and tubing in oil wells. The fabricating characteristics of bessemer seamless pipe have been found to be similar to open-hearth steel. Experience has indicated that the material may be used to replace open-hearth seamless pipe in many standard specifications including A.S.T.M. Specifications A 53 and A 120, API Specifications 5 L, 5 LX, and 5 A and is, therefore, suitable for a wide range of applications.

MR. R. C. FITZGERALD[5] (*by letter*).—The authors have attempted the very difficult task of describing small differences in weldability in terms of well-known mechanical and metallurgical tests, along with less well-known cracking tests. As is usually the case, the cracking tests appear to be more definitive than do the standard tests. The real question of weldability seems to be more adequately defined in terms of such cracking tests as were employed in the present work.

The differences in hardenability between the coarse and fine-grain open-hearth and the fine-grain killed bessemer, as demonstrated on the hardenability chart of Fig. 1, are quite pronounced but it must be recognized that this chart is a single line or average properties chart, whereas the actual values no doubt show considerable overlap in hardness from specimen to specimen. The development of such charts in practical tests to determine weldability have been found very difficult, and in steels of relatively low hardenability the differences are often too small to be adequately demonstrated by such tests. The use of a $\frac{1}{2}$-in. diameter Jominy bar instead of the standard 1-in. diameter bar should make the test more discriminating in these low-hardenability steels. Figure 1 shows the average properties of a number of tests, and the average values do illustrate the relatively slight differences in hardenability that exist.

It is interesting to note that the impact tests over a range of temperatures show very little difference between the open-hearth and bessemer steels, whereas the microstructural differences noted in the heat-affected zones and fusion lines are considerably different. This may be due to the difficulty in accurately locating the notch in the impact specimens.

The authors have stated that under-bead cracking was probably due to the use of an E 6010 type electrode, as well as the differences in hardenability, and that cracking would probably be avoided with all steels tested if a low-hydrogen electrode were used. This is no doubt true; but it is also true that for field welding, or for welding in the horizontal fixed position where the electrode must be manipulated in all welding positions, the low-hydrogen electrode is still extremely difficult to handle, and pipe welding, perforce, must be done with the 10 type electrode coating. Thus, the hardenability of the pipe material to be welded is still of concern to the users of pipe materials. Welding procedures which are designed to produce first quality welds are not always followed in all details and apparently minor deviations in hardenable materials can sometimes produce disastrous results. Such minor procedure deviations often go completely unnoticed unless trouble develops therefrom. For this reason where a choice is possible on the basis of weld hardenability, a material of lesser hardenability is the best choice.

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[5] Senior Engineer, Westport Power Production Station, Consolidated Gas Electric Light and Power Co., Baltimore, Md.

The authors are to be commended on a concise presentation of useful information on the properties of some commonly-used pipe materials. This work indicates better welding qualities for the deoxidized bessemer steels. It should be borne in mind, however, that a choice of materials may not be predicated on weldability alone and that cracking propensity of some steels may be overcome by the proper employment of welding preheat and postheat, whether or not such measures are required by code procedures.

MESSRS. WILDER, KENNEDY, AND CROUCH (*authors*).—The variations in hardenability properties discussed by R. C. Fitzgerald for any one type of steel were very small and did not fall within the range of the other steels. The reason for presenting a curve of the average properties was to simplify an interpretation of the hardenability results. With reference to the ½-in. diameter Jominy bar, it is our opinion that the cooling rate is greater compared with a 1-in. diameter bar. This we believe is due in part to the fact that the effect of air cooling is greater in the ½-in. diameter bar.

It is difficult to determine accurately the impact properties of the over-heated zone in girth welds. Several methods of evaluating these properties have been employed in an effort to overcome the objections. However, the simplest and perhaps the most practical method, at the present time, appears to be that employed by the authors. Structural differences were observed in the several types of steel. Some of these differences are due to carbon content and deoxidation practice. Except in the unaffected base metal of the silicon-killed open-hearth steel at low temperature, we do not believe the differences in impact results observed are significant. The impact properties of the heat-affected zone of the silicon-killed open-hearth steel indicate that the higher values obtained at the lower temperatures may be associated with the beneficial effects of heat treatment during welding.

We are in agreement with Mr. Fitzgerald regarding the use of a lime-coated electrode. In another investigation with similar types of steels, we have found that underbead cracking may be prevented with a low-hydrogen electrode. However, satisfactory welds were more difficult to produce with this more expensive type of electrode.

MR. L. K. STRINGHAM[6] (*by letter*).—My conclusions, as one whose principal interest is welding apparently differ somewhat from one whose principal interest is steel making.

My conclusions from the paper are:

(*a*) 0.15 per cent carbon steel is easier to weld under adverse conditions than 0.25 to 0.30 per cent carbon steel.

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[6] Welding Engineer, Lincoln Electric Co., Cleveland, Ohio.

(*b*) High phosphorus steel is reputed to have low impact values at low temperatures. However, the deoxidized acid-bessemer with 0.075 per cent phosphorus has better impact properties at −20 F. than the deoxidized basic open-hearth steels having a phosphorus content of 0.017 per cent.

(*c*) Years of experience in welding of joints has shown that the root bend test is the most difficult to pass. This paper bears this out in that the root bend specimens were stress relieved in order to pass the root bend test.

Failure in the root bend test normally starts at the fusion zone which is frequently both a metallurgical and physical notch. For this discussion, a metallurgical notch is defined as a notch produced by the difference in grain structure of the weld metal and the heat-affected zone and the difference in tensile strength between that of the weld metal and that of the heat affected zone. The physical notch is an actual change in section which frequently occurs at the root of the weld. This physical notch is particularly common when making the root pass without a backup strip. Therefore, the root bend test is logically the one which is the most troublesome. It should be eliminated, not because it is difficult to pass but because it is an unjust test. Who cares whether the root of a pipe joint will bend. It is not called on to bend. The important point is whether the joint will remain intact under service conditions. This will be determined by the toughness of the joint.

The impact values of this paper indicate that the heat-affected zone is the point of greatest toughness, the weld metal next, and the parent metal the zone of least toughness.

None of the above conclusions are mentioned by the authors. I believe they should be added to those given by the authors to a fine paper.

MESSRS. WILDER, KENNEDY, AND CROUCH (*authors*).—We are in agreement with Mr. L. K. Stringham regarding the bend test requirements of the A.S.M.E. Boiler Code. In order to meet the root bend test requirements, it was necessary in this investigation to stress relieve both the open-hearth and bessemer girth welds. This we understand is not always necessary in the pipe fabricating shop and depends upon the welding technique used and wall thickness of the material.

After a satisfactory welding procedure and operator qualifications have been established, it is important that sound welds with satisfactory penetration be consistently obtained. Bend tests required by the A.S.M.E. Boiler Code rules do not always insure these conditions. Further, the conditions of stress in pipe under operating conditions are not necessarily the same in both the transverse and longitudinal directions, and because of this fact, the tests of a girth weld may be evaluated in a different manner than longitudinal welds. It would be desirable and helpful for all concerned if a study were made in the various pipe fabricating shops throughout the country of the most desirable testing procedure for girth welds.

With reference to Mr. Stringham's conclusions regarding our paper, we would like to offer the following comments: We feel that the bessemer steel, with its lower carbon content, was similar and not superior to the open-hearth steel, with respect to the A.S.M.E. Boiler Code welding tests and our weld bead flattening tests. However, with reference to the weld bead hardness tests, cracking susceptibility tests, microstructure, and hardenability, the bessemer material had more favorable properties due essentially to its lower carbon content. Considering the impact properties, the deoxidized acid-bessemer steel was similar to the silicon-aluminum-killed open-hearth steel and both of these steels were superior at low temperatures to the silicon-killed open-hearth steel. This difference is not associated with steel-making process but more precisely with the method of deoxidizing the steels. The deoxidized acid-bessemer and silicon-aluminum-killed open-hearth steels were made to fine grained deoxidation practice, while the silicon-killed open-hearth steel was made to coarse grained practice.

Considering the phosphorus content of deoxidized acid-bessemer steel, in order to encounter brittleness due to phosphorus, it is, in our opinion, necessary to have much higher phosphorus contents than are permitted in present A.S.T.M. specifications for bessemer seamless pipe. This whole matter of brittleness due to phosphorus in bessemer steel is in many respects a misconception of the actual cause of brittleness in these steels. Mr. Stringham is correct in pointing out that high phosphorus steel is reputed to have low impact values at low temperatures. However, the actual cause of the decrease in toughness may be more precisely explained upon the basis of the deoxidation practice used. If bessemer steel containing up to 0.11 per cent phosphorus is manufactured by the deoxidized acid-bessemer process and properly killed with aluminum, the material will have impact properties similar to regular open-hearth steel. However, if a rimmed, capped, or semi-killed steel-making practice, which is commonly used in the manufacture of regular acid-bessemer steel by the bottom blowing practice is used, a decrease in toughness may be encountered at low temperatures compared with similar open-hearth steels. This difference in toughness between the bessemer and open-hearth steels with similar deoxidation practices is due essentially to the fact that the steels were not thoroughly killed with aluminum or an equivalent type of material.

MR. D. H. COREY[7] (*by letter*).—Is the process by which the deoxidized acid-bessemer steel is made inherently such that the carbon content is always relatively low, that is, in the neighborhood of 0.15 per cent, as reported in the paper, or may it approach the carbon limitation for A.S.T.M. Specifications A 53 grade B pipe? If the latter is the case, how would you expect the physical properties of 0.25 per cent carbon acid-bessemer pipe, particularly the hardenability and tendency toward underbead cracking, to compare with open-hearth pipe of similar carbon content?

MESSRS. WILDER, KENNEDY, AND CROUCH (*authors*).—D. H. Corey raises the question regarding the carbon content of A.S.T.M. Specifications A 53 grade B bessemer seamless pipe. The average carbon content of grade B bessemer seamless pipe is 0.15 per cent carbon and grade B open-hearth seamless pipe 0.25 per cent carbon. The reason for this difference is that A.S.T.M. Specifications A 53 requirements can be met with the lower carbon bessemer steel because of its phosphorus content. Apparently hardenability and underbead cracking are more closely related to carbon content than phosphorus content within the limits studied. This is one of the inherent advantages of bessemer seamless pipe.

In answer to Mr. Corey's question regarding the properties of bessemer seamless pipe with 0.25 per cent carbon, we would expect the hardenability and cracking susceptibility to be greater than open-hearth steel with a similar content, but the tensile properties, particularly the yield strength, would be too high for use under A.S.T.M. Specifications A 53. In other words, the bessemer product would not be similar to the open-hearth product. The physical properties of the two materials should be similar, particularly for fabrication purposes. Although A.S.T.M. Specifications A 53 grade B bessemer seamless has lower hardenability and less cracking susceptibility compared with the open-hearth grade, we do not wish to imply at this time that weldability during fabrication is appreciably different.

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[7] Detroit Edison Co., Detroit, Mich.

MR. JEROME J. KANTER.[8]—Mr. Wilder and his co-authors have based their comparison upon grade B pipe, which is, of course, a pertinent one from two standpoints: one, that grade B is the limitation placed upon the bessemer seamless pipe under A.S.T.M. Specifications A 53 and the second point, I imagine, would be that in the field of design problems where one must use grade B pipe, the considerations which he has developed are the pertinent ones.

Thus we have open-hearth pipe at the 0.25 per cent carbon level compared with the bessemer pipe at the 0.14 per cent carbon level, and it is not surprising that in such comparison the hardenability of the open-hearth steel would show up as greater than the hardenability of bessemer steel. However, in the case of open-hearth pipe, one has the alternative of using grade A, and presumably grade A pipe might be obtained at the 0.14 per cent carbon level also, so the designer who chooses to use open-hearth pipe has the alternative of designing either with grade A or grade B.

In view of that alternative, I feel that the engineering value of the comparison would be greatly enhanced if the studies were extended to cover the welding properties of grade A open-hearth pipe. That would mean selecting some heats of open-hearth pipe in the lower carbon range and also developing their properties.

MESSRS. WILDER, KENNEDY AND CROUCH (*authors*).—Mr. Kanter has suggested grade B bessemer and open-hearth seamless pipe be compared with grade A open-hearth seamless pipe. This would, as Mr. Kanter points out, enhance the engineering value of the comparison, as under certain conditions the designer may choose to use grade A material which is only made from open-hearth steel. We plan to develop additional information concerning grade A seamless pipe and wish to express our appreciation for this suggestion.

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[8] Materials Research Engineer, Crane Co., Chicago, Ill.

WELDS BETWEEN DISSIMILAR ALLOYS IN FULL-SIZE STEAM PIPING*

By R. U. Blaser,[1] F. Eberle,[1] and J. T. Tucker, Jr.[1]

Synopsis

Factors affecting the safe operation of ferritic-austenitic weld joints in large-size steam piping were studied by means of a simulated proof test.

The specimen consisted of a 10$\frac{3}{4}$-in. diameter section of 2$\frac{1}{4}$ Cr - 1 Mo[2] steam piping welded to suitably designed and dimensioned end pieces of 18 Cr - 8 Ni Cb wrought and 16 Cr - 13 Ni - 2 Mo Cb cast materials, using 19 Cr - 9 Ni Cb welding electrodes. Cracks were detected by radiographic means in the 2$\frac{1}{4}$ Cr - 1 Mo pipe at the edge of the weld joint after 4631 hr. at 1100 F. and 1500 psi. pressure, involving 47 week-end shutdowns to atmospheric conditions.

Metallurgical examination showed that the principal factors contributing to the failure were stresses up to yield strength due to differences in thermal expansion of the ferritic and austenitic materials, accelerated creep, intergranular stress-oxidation and weakening of the ferritic metal adjacent to the fusion zone due to carbon migration into the austenitic weld metal.

The general trend in modern steam plants is toward increased pressures and higher temperatures which, in turn, require thicker sections in piping, valves and structural members where strength is a major consideration. In order to minimize resulting fabrication difficulties, weight, and costs, materials of greater load-carrying ability at elevated temperatures are constantly being sought. This has led to the use of various austenitic high chromium-nickel alloys which differ from the lower strength ferritic alloys in some important physical properties such as thermal conductivity, coefficient of expansion, modulus of elasticity, and others.

The joining of such dissimilar austenitic and ferritic alloys presents important design and fabrication problems. Mechanical joints for high temperatures and pressures are large and cumbersome and have inherent difficulties such as stud relaxation and gasket leakage. Welded joints are therefore desirable but offer other problems due to differences in the physical and metallurgical characteristics of the ferritic and austenitic materials.

The difference in the high-temperature load-carrying ability of the two types of materials calls for different thicknesses and special design considerations. The differences in thermal conductivity and in the coefficient of expansion set up stresses at the joint which can be of considerable magnitude and which become of great importance if the operation includes a large number of shutdowns or cycles of temperature and pressure.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Chief, Engineering Physics Section, Chief Metallurgical Engineer, and Senior Test Engineer, respectively, Research and Development Dept., The Babcock & Wilcox Co., Alliance, Ohio.

[2] 2$\frac{1}{4}$ per cent chromium, 1 per cent molybdenum alloy steel; additional elements are indicated by the chemical symbols Cb, Ti, etc.

Microstructural changes during exposure at elevated temperatures may also have a deleterious effect upon the weld joint properties.

PURPOSE OF TESTS

In order to explore the effect of some of the factors which may affect the safety of ferritic-austenitic weld joints in steam piping, a full-size proof test was conducted. The test procedure involved maintaining the specimen at 1100 F. and 1500 psi. internal pressure with periodic shutdowns as described subsequently.

TEST SPECIMEN

The test specimen which is shown in Fig. 1 represents the steam piping used on many installations. The three base materials employed were 18 Cr-8 Ni Cb, $2\frac{1}{4}$ Cr-1 Mo; and a cast material of 16 Cr-13 Ni-2 Mo Cb. This latter material was included because of its frequent use for large steam valves. The pipe thick-

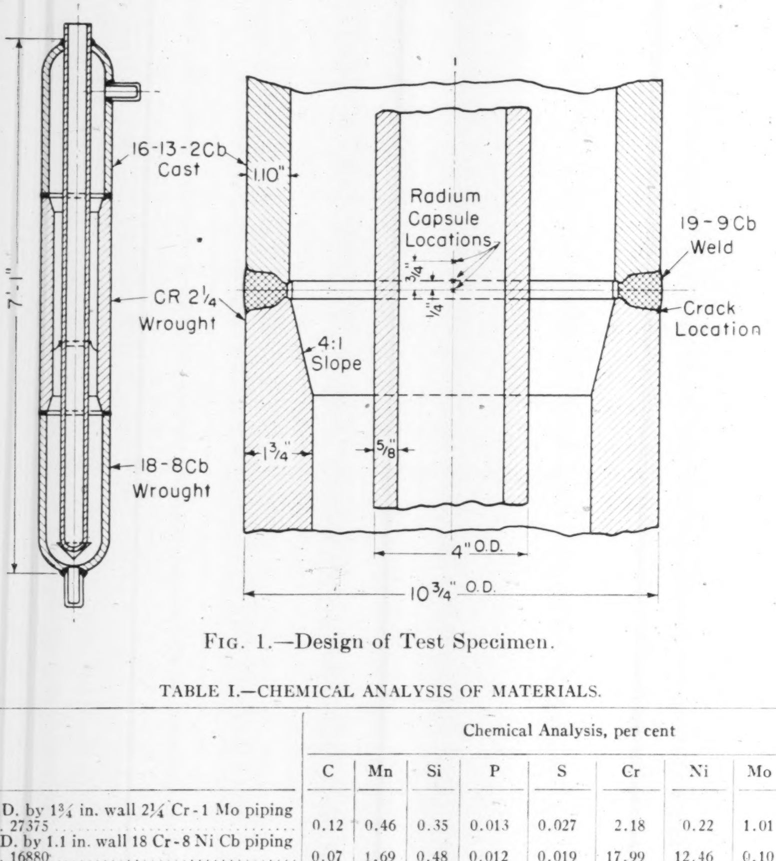


FIG. 1.—Design of Test Specimen.

TABLE I.—CHEMICAL ANALYSIS OF MATERIALS.

	Chemical Analysis, per cent								
	C	Mn	Si	P	S	Cr	Ni	Mo	Cb
$10\frac{3}{4}$ in. O.D. by $1\frac{3}{4}$ in. wall $2\frac{1}{4}$ Cr-1 Mo piping heat No. 27375	0.12	0.46	0.35	0.013	0.027	2.18	0.22	1.01	...
$10\frac{3}{4}$ in. O.D. by 1.1 in. wall 18 Cr-8 Ni Cb piping heat No. 16880	0.07	1.69	0.48	0.012	0.019	17.99	12.46	0.10	0.96
$10\frac{3}{4}$ in. O.D. by 1.1 in. wall 16 Cr-13 Ni-2 Mo Cb casting heat No. 26383	0.10	0.74	0.71	....	....	15.51	13.74	1.89	1.05
Weld metal, 18 Cr-8 Ni Cb to $2\frac{1}{4}$ Cr-1 Mo weld junction	0.07	1.87	0.71	0.024	0.010	19.17	10.32	....	0.88
Weld metal, 16 Cr-13 Ni-2 Mo Cb to $2\frac{1}{4}$ Cr-1 Mo weld junction	0.07	1.68	0.55	0.023	0.010	18.87	10.50	....	0.95
Internal tube, 4 in. O.D. by 0.625 in. wall 16 Cr-13 Ni-3 Mo Cb	0.07	1.56	0.52	0.011	0.017	17.26	14.80	2.25	0.70

nesses were chosen to give working stresses in the circumferential direction of 3900 psi. for the $2\frac{1}{4}$ Cr-1 Mo, and 6600 psi. for both the 18 Cr-8 Ni Cb and the 16 Cr-13 Ni-2 Mo Cb cast material. The resultant thicknesses were $1\frac{3}{4}$ in. for the $2\frac{1}{4}$ Cr-1 Mo center section and 1.10 in. for the stainless sections. The chemical composition of the materials involved is given in Table I, and their thermal history is described in Table II.

A central tube shown in Fig. 1 was made of columbium stabilized 16 Cr-13 Ni-3 Mo material and contained an internal electric heating element. This permitted the pipe to be heated from the inside, thereby simulating operating conditions. A spacer was tack-welded into place to center the internal tube for best heat distribution to the specimen. Two stub connections of $2\frac{1}{2}$ in. pipe size were provided for contemplated steam cooling in a second phase of the program. A third phase was a water quench through these same connections. It was for this water-quench test that a thin sheet-metal cone was welded at the closed end of the internal tube to prevent extreme thermal shock on this piece.

TABLE II.—THERMAL HISTORY OF MATERIALS.

10 ¾ in. O.D. by 1¾ in. wall 2¼ Cr-1 Mo piping	Heated slowly to 1500–1525 F., held for 12 hr. and slowly cooled in furnace to 600 F.
10¾ in. O.D. by 1.1 in. wall 18 Cr-8 Ni Cb piping	Annealed at 2100 F., air cooled, reheated to 1750 F., held for 1 hr. and air cooled.
10¾ in. O.D. by 1.1 in. wall, 16 Cr-13 Ni-2 Mo Cb cast piping	Annealed for 10 hr. at 1904–1994 F., air cooled to 750 F., reheated to 1560–1650 F., held for 10 hr. and furnace cooled to 500 F.

Electrodes used in making the test welds were of 19 Cr-9 Ni Cb composition. The weld groove design is shown in Fig. 1. The $2\frac{1}{4}$ Cr-1 Mo backing ring was tack-welded in place and the weld completed with nine layers of weld metal, using $\frac{1}{8}$-in. and $\frac{5}{32}$-in. diameter electrodes. The total number of beads in each weld was about sixteen. The sections being joined were held at a minimum temperature of 400 F. during the welding. No post-weld treatment was applied. The welds were ground to a smooth contour and radiographed.

TEST APPARATUS AND INSTRUMENTATION

Figure 2 shows the apparatus used in testing the specimen. In the background is a small, vertical electric boiler which supplies the steam and pressure for the specimen. This boiler consisted of a section of pipe around which electric resistance heating coils were wound; insulating material and an outer jacket completed the assembly. This steam-pressure system was equipped with a safety valve, gage glass for water level observation, pressure gages, and various vents and drain valves. The pressure was maintained at 1500 psi. by the Bristol pressure controller shown on the instrument panel.

The test temperature was obtained with a 15-kw. electric heating unit inserted into the inner tube of the specimen and was controlled by a thermocouple at the heater in conjunction with the Leeds & Northrup controller-recorder shown on the panel board. Temperatures were measured at various locations along the specimen, as well as in the heater itself.

Figure 3 is a top view of the specimen in its removable insulated guard and shows the baffle plates which prevented large convection currents in the 1-in. space between the guard and the specimen. Aluminum foil was used around the bottom half of the specimen and on the lower internal surface of the guard. These provisions gave a temperature gradient at the welds no greater than 10 F. over a 5-in. band containing the weld and no greater than 2 F. over a 2-in. band containing the weld. Circumferential temperature differences were within 4 F.

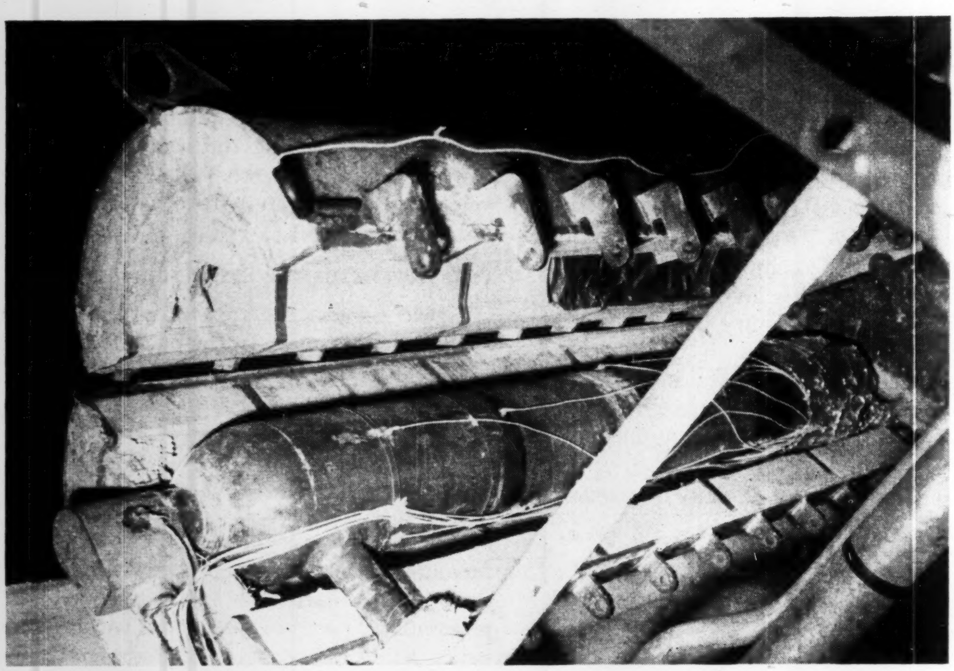


FIG. 3.—Top View of Specimen in Removable Insulated Guard.

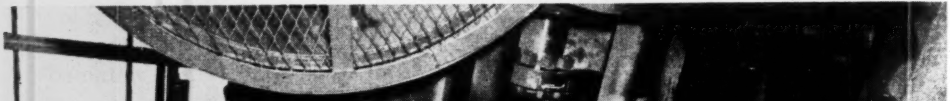


FIG. 2.—Apparatus for Testing the Specimen.

The insulated guard was hinged and constructed in the manner shown so that it could be removed following each shutdown to permit close inspection. The complete assembly was surrounded by blasting mats as a safety precaution.

TEST PROCEDURE

The procedure followed was to keep the specimen at 1100 F. and 1500 psi. pressure 24 hr. a day during the week. On Saturday mornings the heater was turned off and the pressure vented to the atmosphere. The top blasting mat was then lifted and the insulated guard removed from the specimen so that more rapid cooling would occur. On Monday mornings the weld junctions were given a careful examination which included both, visual inspection and the use of fluorescent liquid technique. Following this, the guard was replaced and another heating cycle begun. The steam pressure from the boiler was not connected to the specimen until the latter had reached saturation temperature for the boiler pressure. These heating-and-cooling cycles took about 7 hr. each as shown in Fig. 4. On every fourth shutdown a radiographic inspection with a radium capsule was also used.

TEST RESULTS

After a total time at temperature of 4631 hr., defects were detected by radiographic observation at the locations shown in Fig. 1. Several positions of the radium capsule were used to determine the location of the cracks.

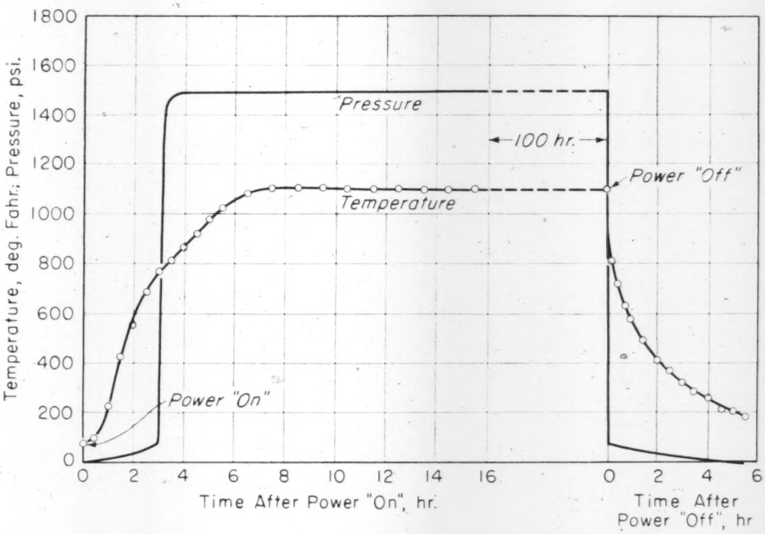


FIG. 4.—Heating and Cooling Cycles.

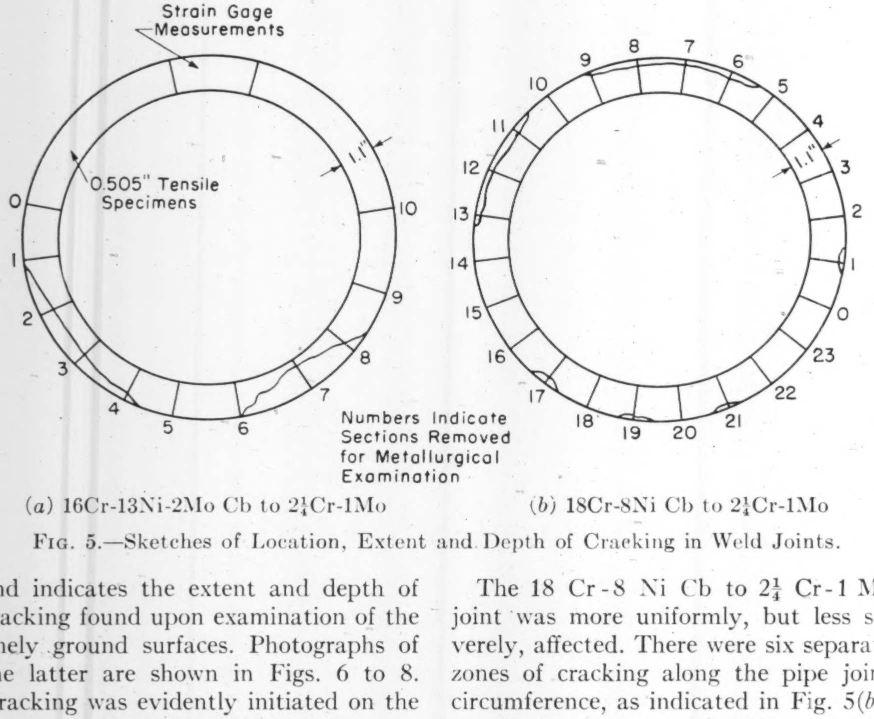
The test life of the specimen included 47 complete temperature and pressure cycles as described. In addition, several partial or complete temperature or pressure cycles occurred because of failures of the auxiliaries.

These unintentional shutdowns resulted in pressure or temperature gradients in no case more severe than the regular cycles.

The fluorescent liquid technique picked up the crack only after the surface oxide at the weld metal junction was completely removed. This was not done completely during each weekly cycle in order to avoid the formation of a mechanical surface notch by the localized grinding operation.

METALLURGICAL EXAMINATION

The two austenitic-ferritic weld junctions were subsequently cut out and sectioned across the weld into a number of slices to determine the location, mode, and magnitude of the failure. Figure 5 presents the layout of these test sections and indicates the extent and depth of cracking found upon examination of the finely ground surfaces. Photographs of the latter are shown in Figs. 6 to 8. Cracking was evidently initiated on the outside surface of the $2\frac{1}{4}$ Cr-1 Mo pipe close to the interface with the 19 Cr-9 Ni Cb weld metal and propagated inwardly, following the fusion zone closely. Cracking was not continuous along the circumference of the pipe joint: There were only two zones in the 16 Cr-13 Ni-2 Mo Cb weld joint where failure occurred, one extending from about location 1 to location 4 along a circumferential distance of approximately 3 in., and the second, about $1\frac{1}{2}$ to 2 in. long, from location 6 to location 8 (Fig. 5*a*). The two zones of cracking were about 3 in. apart and were located on the same half of the pipe circumference. The maximum crack penetration in the longer zone was $\frac{3}{8}$ in. and in the shorter zone $\frac{7}{8}$ in. A sample taken from the latter was opened so as to expose the fracture surface which was oxidized and presented a scalloped appearance reflecting the weld bead contours (Fig. 9).



(*a*) 16Cr-13Ni-2Mo Cb to $2\frac{1}{4}$Cr-1Mo (*b*) 18Cr-8Ni Cb to $2\frac{1}{4}$Cr-1Mo

FIG. 5.—Sketches of Location, Extent and Depth of Cracking in Weld Joints.

The 18 Cr-8 Ni Cb to $2\frac{1}{4}$ Cr-1 Mo joint was more uniformly, but less severely, affected. There were six separate zones of cracking along the pipe joint circumference, as indicated in Fig. 5(*b*). Crack penetration was uniformly shallow, ranging from $\frac{3}{32}$ to $\frac{3}{16}$ in.

Microscopic examination of various samples removed from cracked and crack-free sections of the two dissimilar weld joints confirmed that cracking was initiated in the $2\frac{1}{4}$ Cr-1 Mo pipe surface close to and adjacent to the weld metal interface. During the high-temperature part of the temperature cycle, the

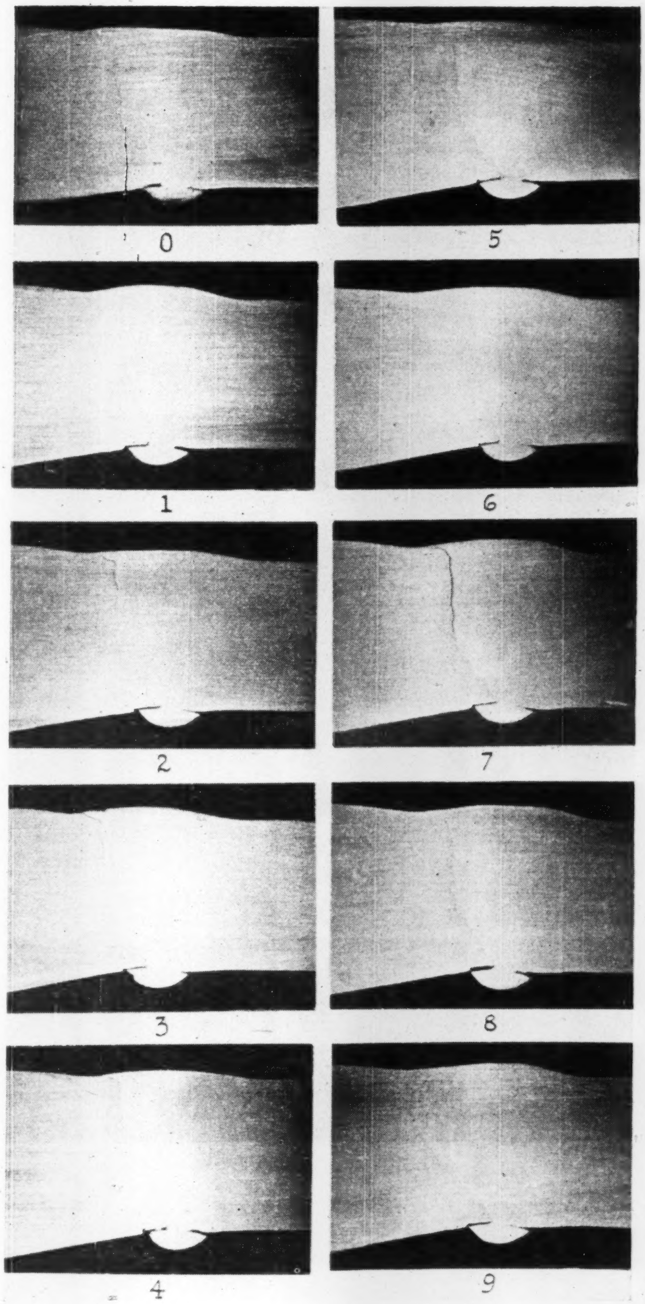


FIG. 6.—Cross-Sections of 16Cr-13Ni-2Mo Cb (right) to $2\frac{1}{4}$Cr-1Mo (left) Weld Joint, Showing Occurrence and Extent of Cracking in $2\frac{1}{4}$Cr-1Mo Pipe.

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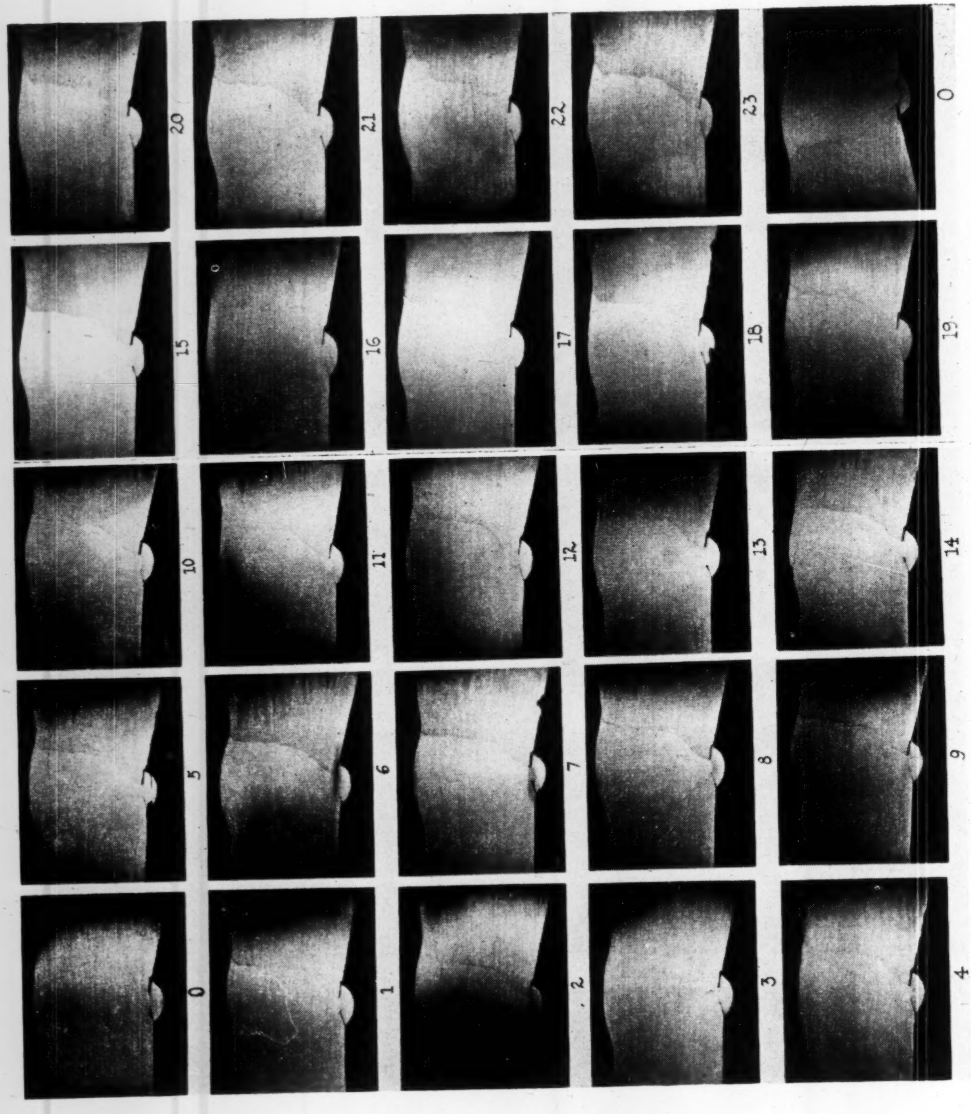
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FIG. 7.—Cross Section of 18Cr-8Ni-Cb (left) to 2¼Cr-1Mo (right) Weld Joint, Showing Occurrence and Ex-

BINDING

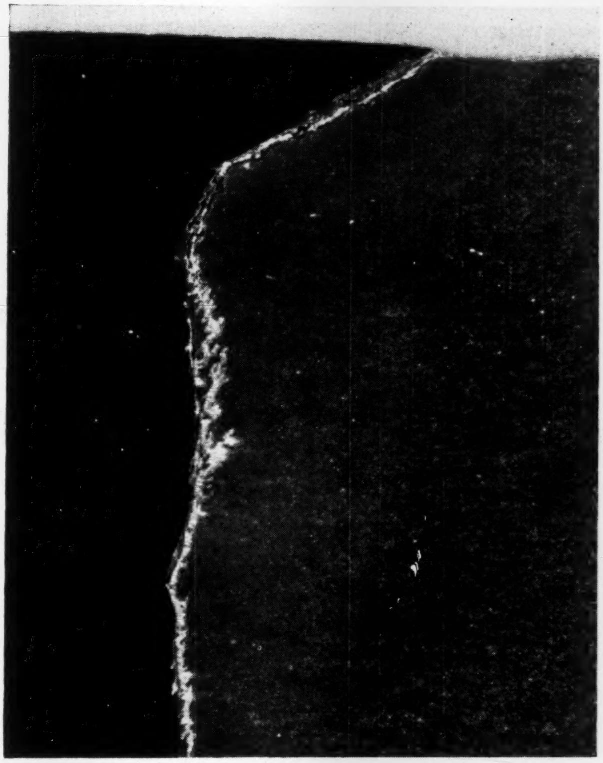


FIG. 8.—**Section Through 16Cr-13Ni-2Mo Cb to $2\frac{1}{4}$Cr-1Mo Weld Joint Between Locations 7 and 8 (×20).**

Surface micropolished and etched with alkaline sodium chromate to reveal dissolved oxygen in cracked zone.

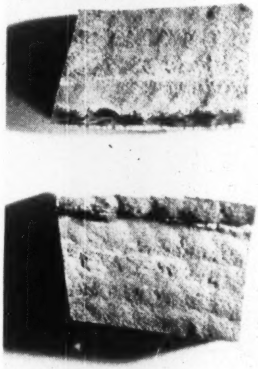
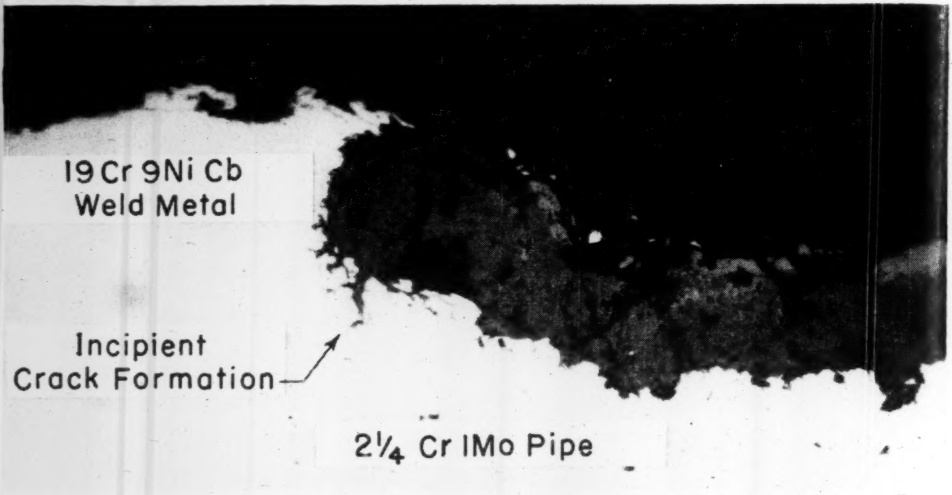


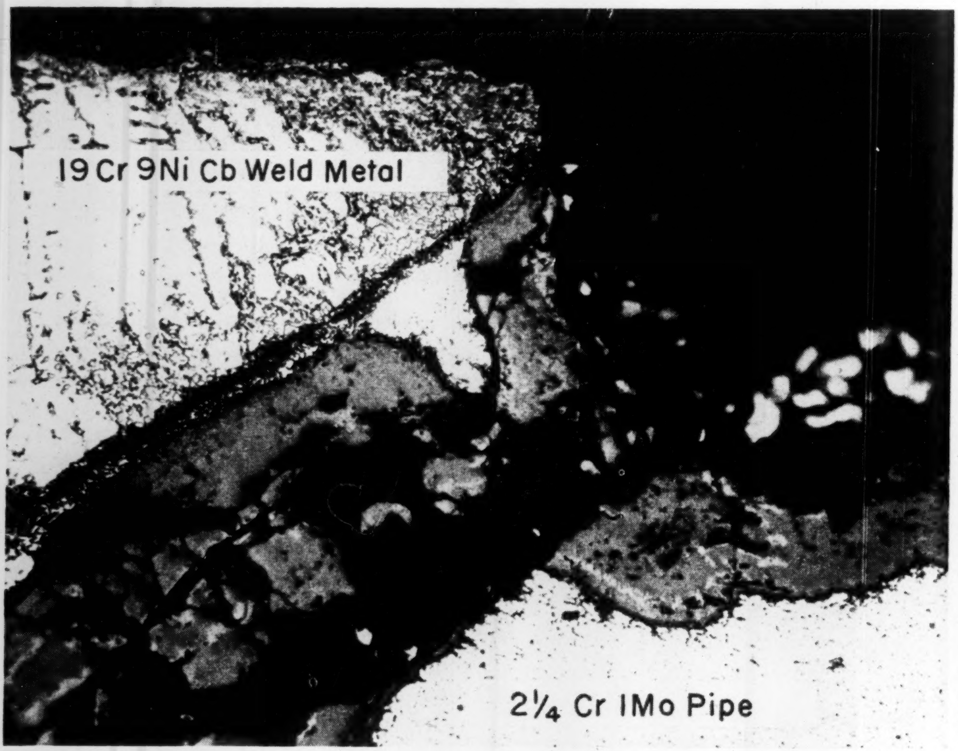
FIG. 9.—Fracture Surface of Sample Removed at Location 7 of 16Cr-13Ni-2Mo Cb to $2\frac{1}{4}$Cr-1Mo Weld Joint, Showing Weld Bead Contours.

$2\frac{1}{4}$Cr-1Mo material scaled readily and formed an abrupt step-like change from the 19 Cr-9 Ni Cb weld metal surface to the $2\frac{1}{4}$ Cr-1 Mo pipe surface (Fig. 10). This abrupt change undoubtedly promoted stress concentration and concomitant accelerated localized oxidation of the $2\frac{1}{4}$ Cr-1 Mo material, leading to the development of an oxide notch at the highly stressed weld junction. The formation of intergranular oxide notches in the $2\frac{1}{4}$ Cr-1 Mo pipe surface away from the weld where only operational and thermal stresses were effective was quite general and testified to the fact that at the given test temperature the oxidation resistance of the material began to fall off (Fig. 11).

Under the combined influence of stress concentration and oxidation, the stress-

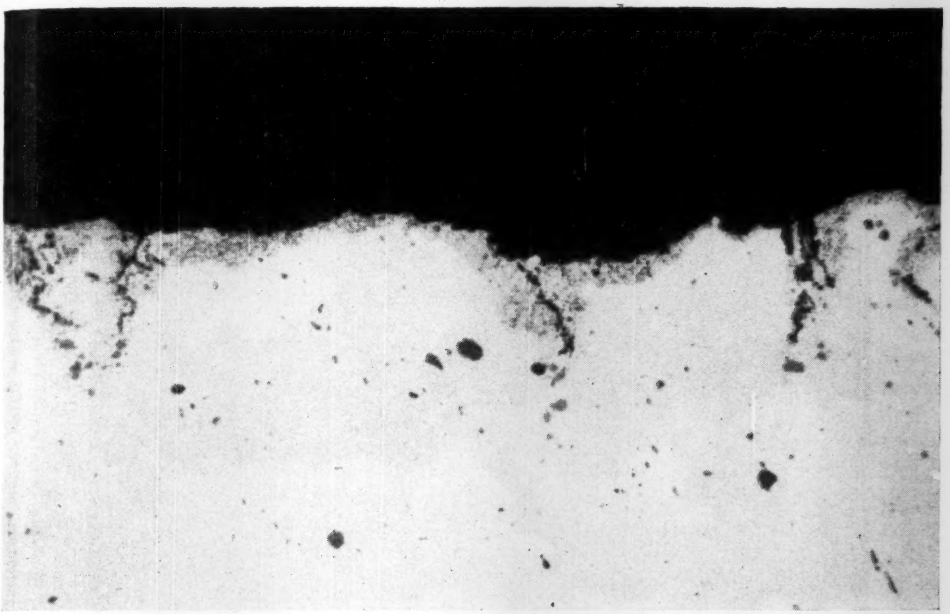

19 Cr 9Ni Cb
Weld Metal
Incipient
Crack Formation
2¼ Cr 1Mo Pipe

Unetched (×500)

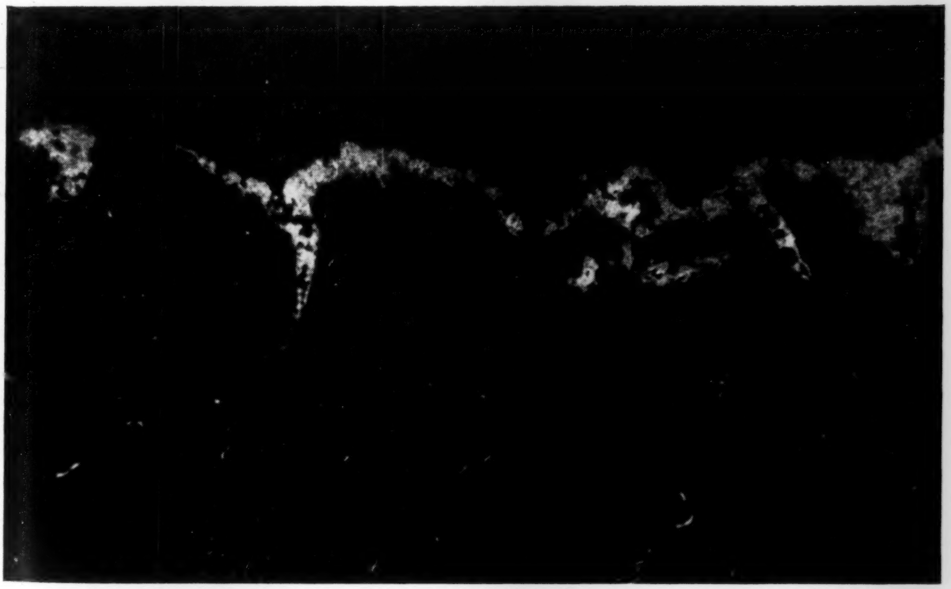

19 Cr 9Ni Cb Weld Metal
2¼ Cr 1Mo Pipe

Picral Etch (×1000)

Fig. 10.—Sections Through Crack Free (Top) and Cracked (Bottom) Weld Junctions, Showing Preferential Scaling of 2¼Cr-1Mo Pipe Surface and Notch Formation at Weld Junction.

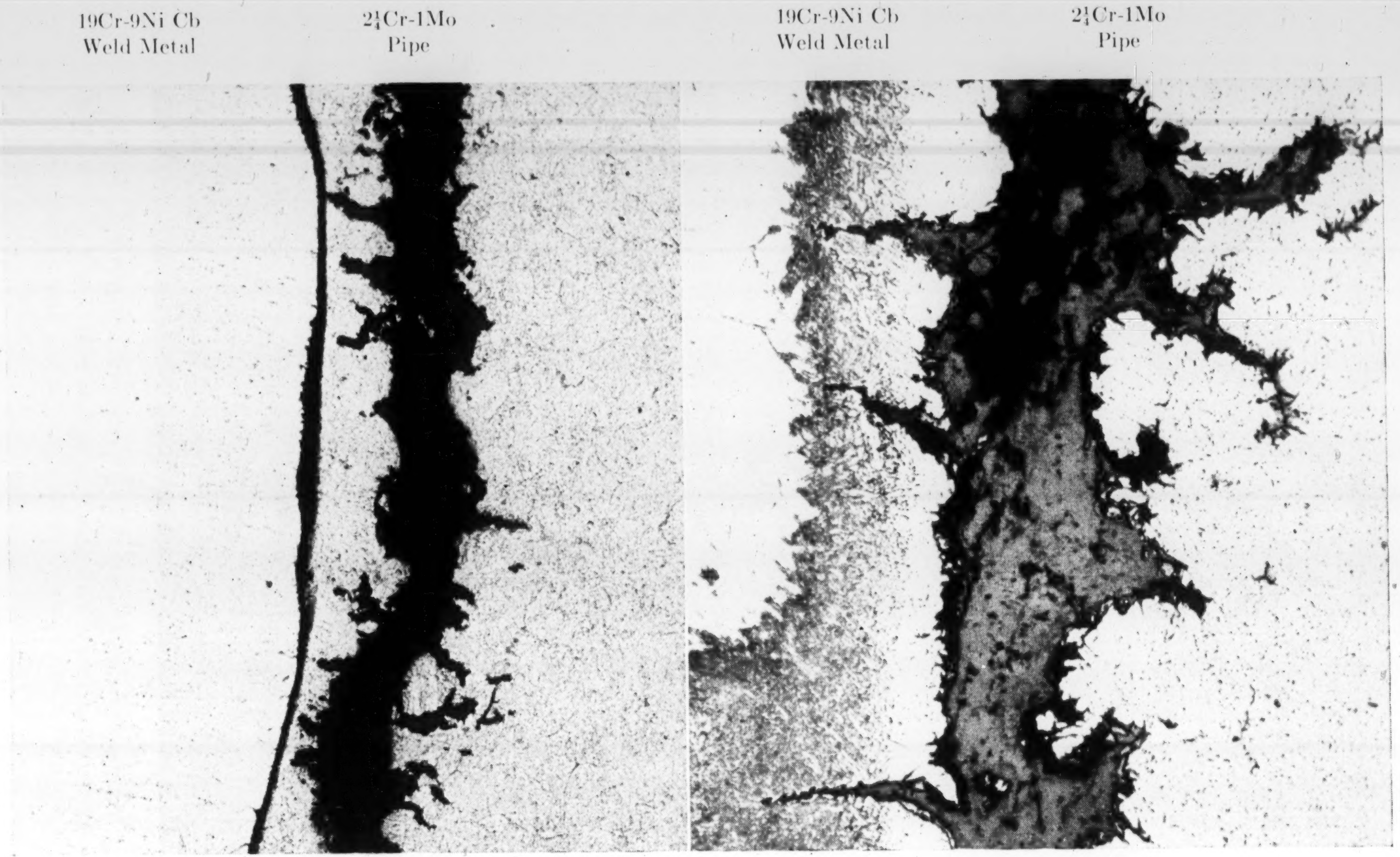


Picral Etch (×1000)



Alkaline Sodium Chromate Etch Revealing Dissolved Oxygen (×1000)

FIG. 11.—Photomicrographs of $2\frac{1}{4}$Cr-1Mo Pipe Surface 2 in. from Edge of Weld, Showing Incipient Intergranular Oxidation and Surface Notch Formation.


19Cr-9Ni Cb
Weld Metal
2¼Cr-1Mo
Pipe
19Cr-9Ni Cb
Weld Metal
2¼Cr-1Mo
Pipe

Picral Etch (×100)

Picral Etch (×500)

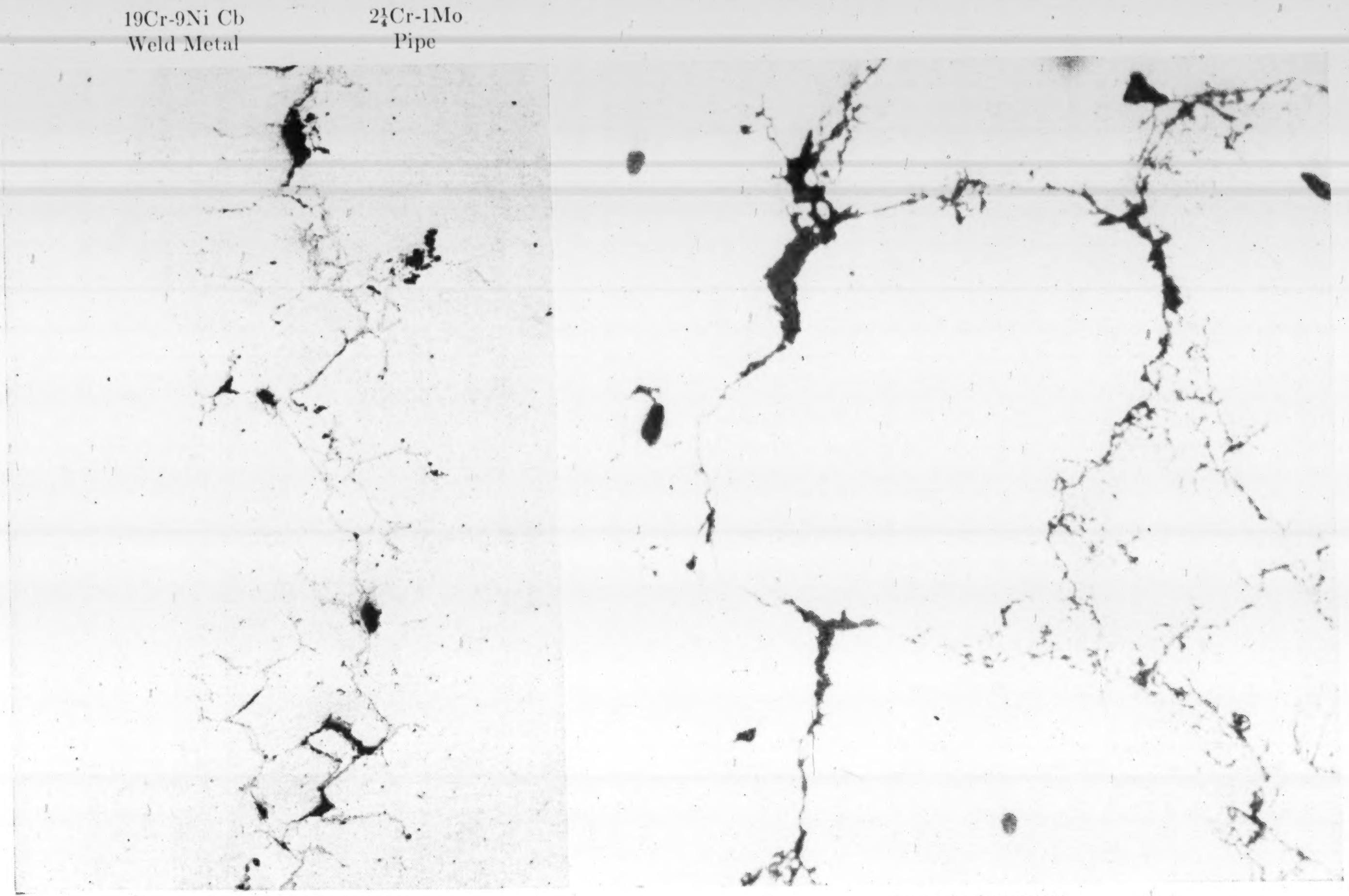
FIG. 12.—Main Crack in 16Cr-13Ni-2Mo Cb to 2¼Cr-1Mo Weld Joint, Showing Location of Crack in 2¼Cr-1Mo Pipe.

Note intercrystalline type of failure and carbon migration to weld junction and into 19Cr-9Ni Cb weld metal.

raising oxide notch which formed in the $2\frac{1}{4}$ Cr-1 Mo pipe surface adjacent to the weld metal junction developed into a crack which propagated inwardly in an intercrystalline manner, always following the fusion zone at a close distance and frequently branching out in all directions (Fig. 12). It is important to note that the cracks were entirely located in the $2\frac{1}{4}$ Cr-1 Mo base metal. Cracking seems to have been preceded by intergranular oxidation, indicated by the presence of dissolved oxygen and finely precipitated oxides in and at the grain boundaries surrounding the main and branch cracks (Figs. 13 and 14). The degree to which oxidation was involved in this failure is illustrated by Fig. 15 which shows the microstructure of the polished and etched fracture surface at location 7 of the 16 Cr-13 Ni-2 Mo Cb weld joint. Evidently, the extent of oxidation was much greater in the plane perpendicular to the pipe axis than in the plane parallel to the latter. It is fairly obvious that a very significant part of the sound metal in the most highly stressed narrow zone adjacent to the weld junction has been converted into brittle oxide, reducing its effective cross-section.

A study of the microstructure of the affected region indicated that some carbon depletion had occurred. The photomicrographs presented in Fig. 16 show that much of the carbide clearly visible in the weld-unaffected $2\frac{1}{4}$ Cr-1 Mo parent metal has disappeared in the zone containing the crack, leaving the molybdenum-containing intracrystalline precipitate behind which forms at temperatures in the vicinity of that used in the present test. Carbon seems to have concentrated in the fusion zone, and there is also evidence that some of this carbon has diffused into the 19 Cr-9 Ni Cb weld metal (Fig. 17). These observations were confirmed by the results of a microhardness survey of the weld junction, which showed a dip in hardness in the $2\frac{1}{4}$ Cr-1 Mo pipe metal adjacent to the fusion line, a steep hardness rise in the latter, and a notable hardness increase in the 19 Cr-9 Ni Cb weld metal adjacent to the junction line (Fig. 18).

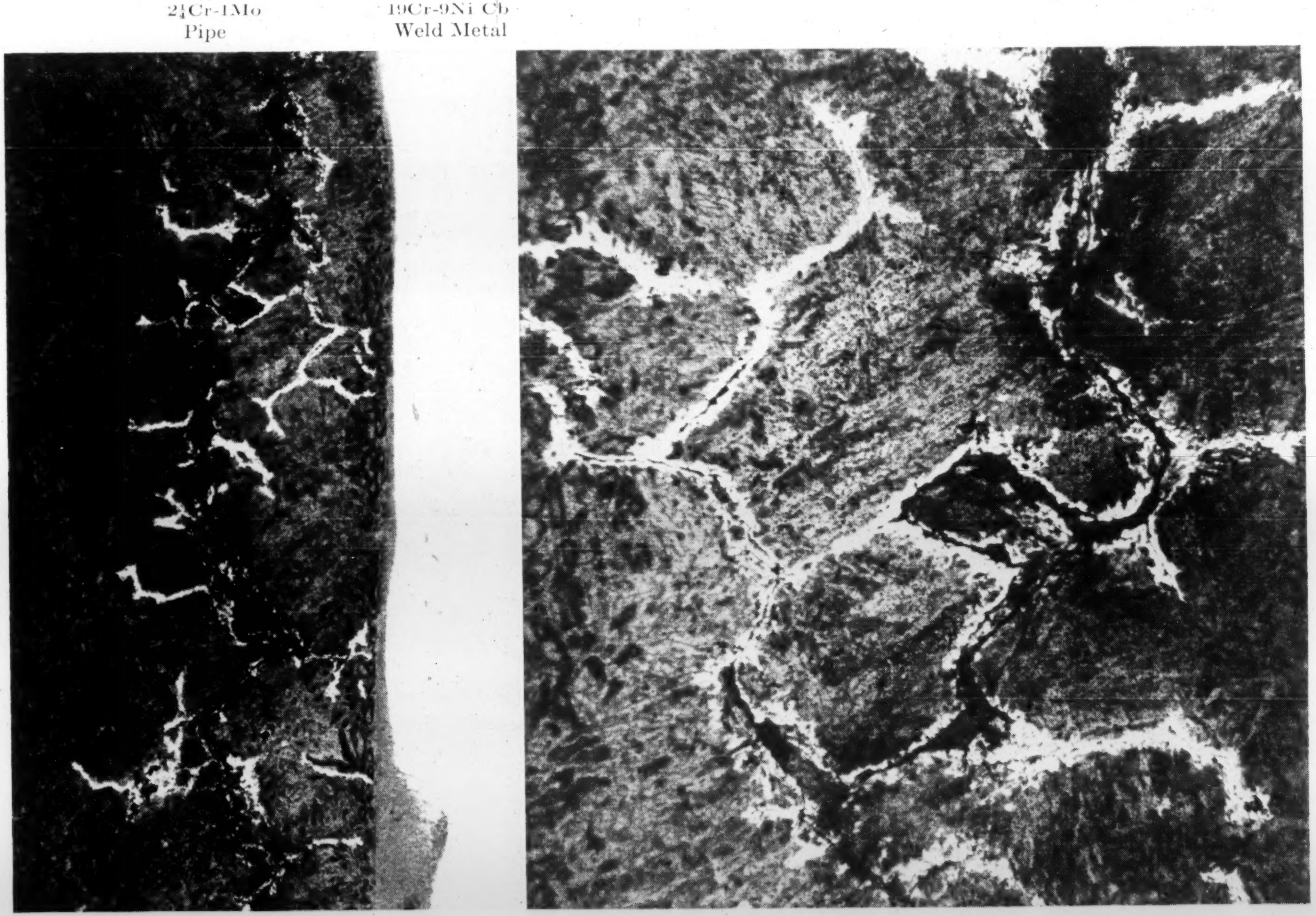
In order to obtain an indication of the effect of the described microstructural changes upon the mechanical properties of the weld joint, transverse weld tension specimens were prepared from a crack-free section of the 16 Cr-13 Ni-2 Mo Cb to $2\frac{1}{4}$ Cr-1 Mo weld joint and tested at room temperature and at 1100 F. For comparison, another dissimilar weld was made with a $2\frac{1}{4}$ Cr-1 Mo forging and an 18 Cr-8 Ni Cb forging and also tested at room temperature, but in the as-welded condition. The results of these test are shown in Table III. The comparison specimen, representing the as-welded condition, ruptured in the 18 Cr-8 Ni Cb base metal, probably due to the fact that the $2\frac{1}{4}$ Cr-1 Mo forging had not been annealed prior to the welding. However, it is interesting to note that in this test the weld-affected $2\frac{1}{4}$ Cr-1 Mo base metal displayed a remarkable strength. In contrast to this observation, fracture in the specimens removed from the cycled test pipe occurred in all cases at yield and strength values far below those generally obtained and in a brittle manner. Subsequent microscopic examination revealed that failure took place at the same location at which the large pipe had developed cracks, that is, in the carbon-depleted zone adjacent to the 19 Cr-9 Ni Cb weld metal (Fig. 19). The path of the fracture was, in all cases, entirely transcrystalline. Etching with alkaline sodium chromate showed complete absence of dissolved oxygen in the zone of rupture. No microstructural deteriorations other than carbon migration were noted.


19Cr-9Ni Cb
Weld Metal
2¼Cr-1Mo
Pipe

Light Picral Etch (×100)

Unetched (×1000)

FIG. 13.—Intergranular Oxide Network in Most Highly Stressed Zone of 2¼Cr-1Mo Pipe Adjacent to Fusion Interface (16Cr-13Ni-2Mo Cb to 2¼Cr-1Mo Weld Joint).


$2\frac{1}{4}$Cr-1Mo Pipe
19Cr-9Ni Cb Weld Metal

Alkaline Sodium Chromate Etch (×100)

Alkaline Sodium Chromate Etch (×500)

FIG. 14.—Transverse Weld Section Through Cracked Zone, Polished and Etched to Reveal Dissolved Oxygen (White Areas) in and at Grain Boundaries of $2\frac{1}{4}$Cr-1Mo Pipe Adjacent to Weld Metal.

DISCUSSION

A complete stress analysis of a dissimilar weld joint as described in this paper is quite involved and has not yet been completed. However, the presented observations indicate that the following stress conditions existed:

1. Residual stresses from the welding operation.

2. Operational stresses (circumferential and longitudinal) due to internal pressure. These stresses vary with pressure changes.

3. Stresses resulting from the differential expansion of the ferritic and austenitic materials. These stresses vary in magnitude with temperature changes and are at a maximum just after a major temperature change.

4. Stresses resulting from differences in the thermal conductivities of the ferritic and austenitic materials. These stresses exist only during the heating-and-cooling cycles and disappear at stable operating and at atmospheric conditions.

The combined stresses conceivably span a range of stresses of a magnitude larger

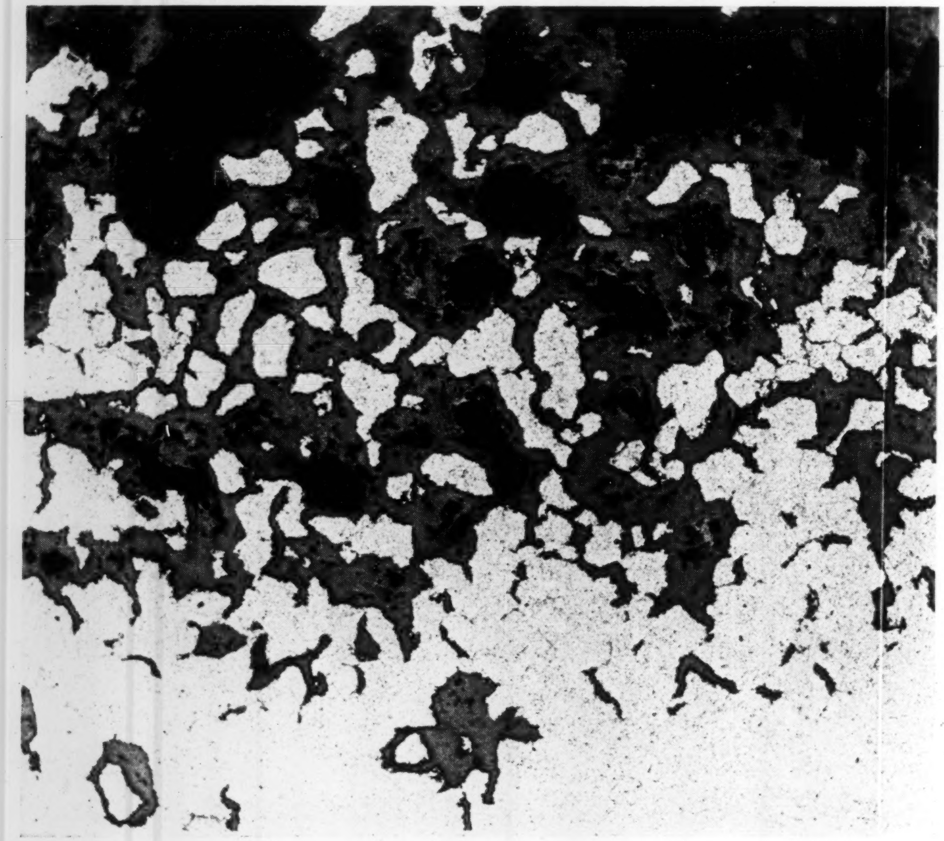


FIG. 15.—Polished and Etched Fracture Surface at Location 7 of 16Cr-13Ni-2Mo Cb to $2\frac{1}{4}$Cr-1Mo Weld Joint, Showing Extent of Oxidation in Plane Perpendicular to Pipe Axis. Etched in Picral (×100).

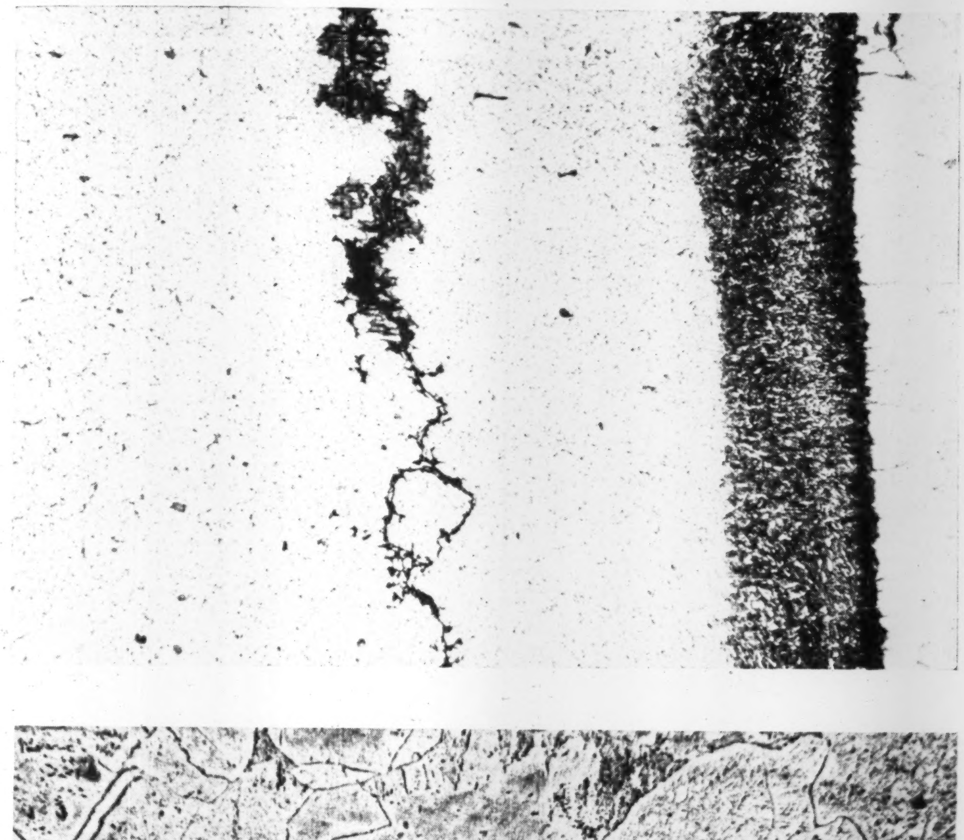


Fig. 16.—Carbide Structure of $2\frac{1}{4}$Cr-1Mo Pipe Near Fusion Zone (Top) and in Weld-Unaffected Base Metal (Bottom). Etched in Picral (×250).

Note absence of Grain boundry Carbide in $2\frac{1}{4}$ Cr-1Mo metal adjacent to fusion line and carbide concentration in latter.

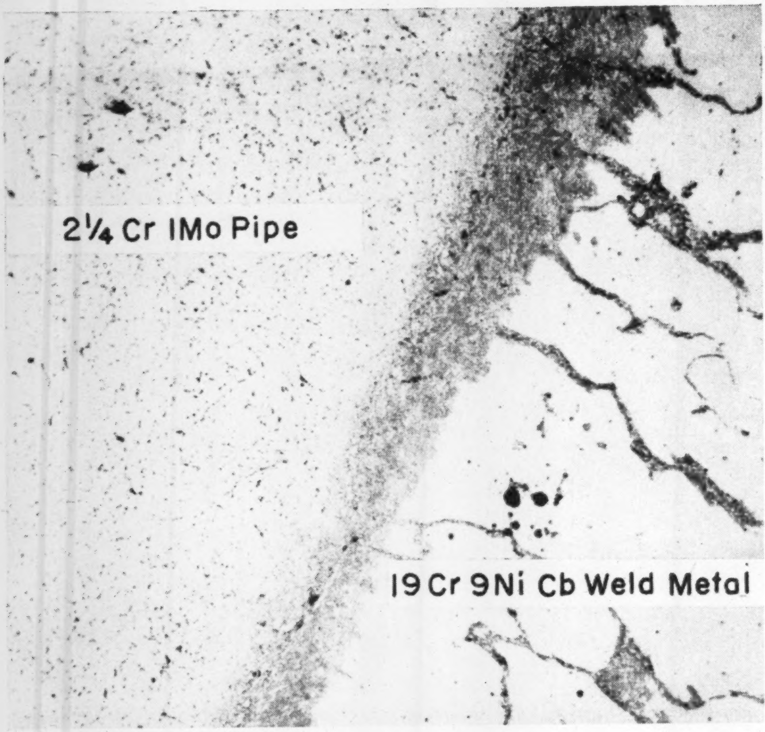

2¼ Cr 1Mo Pipe
19 Cr 9Ni Cb Weld Metal

FIG. 17.—Junction of 2¼Cr-1Mo Pipe and 19Cr-9Ni Cb Weld Metal, Showing Carbide Concentration in Fusion Zone and Carbon Migration Along Grain Boundaries Into the Austenitic Weld Metal. Etched in Picral (×500).

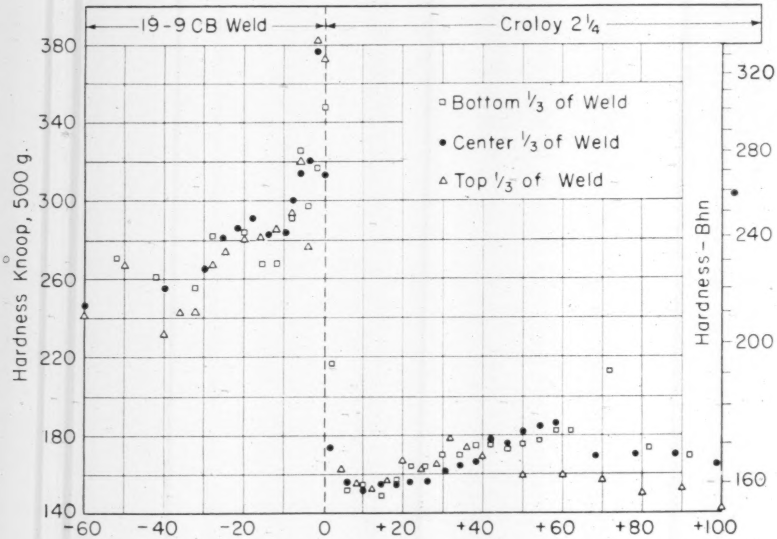
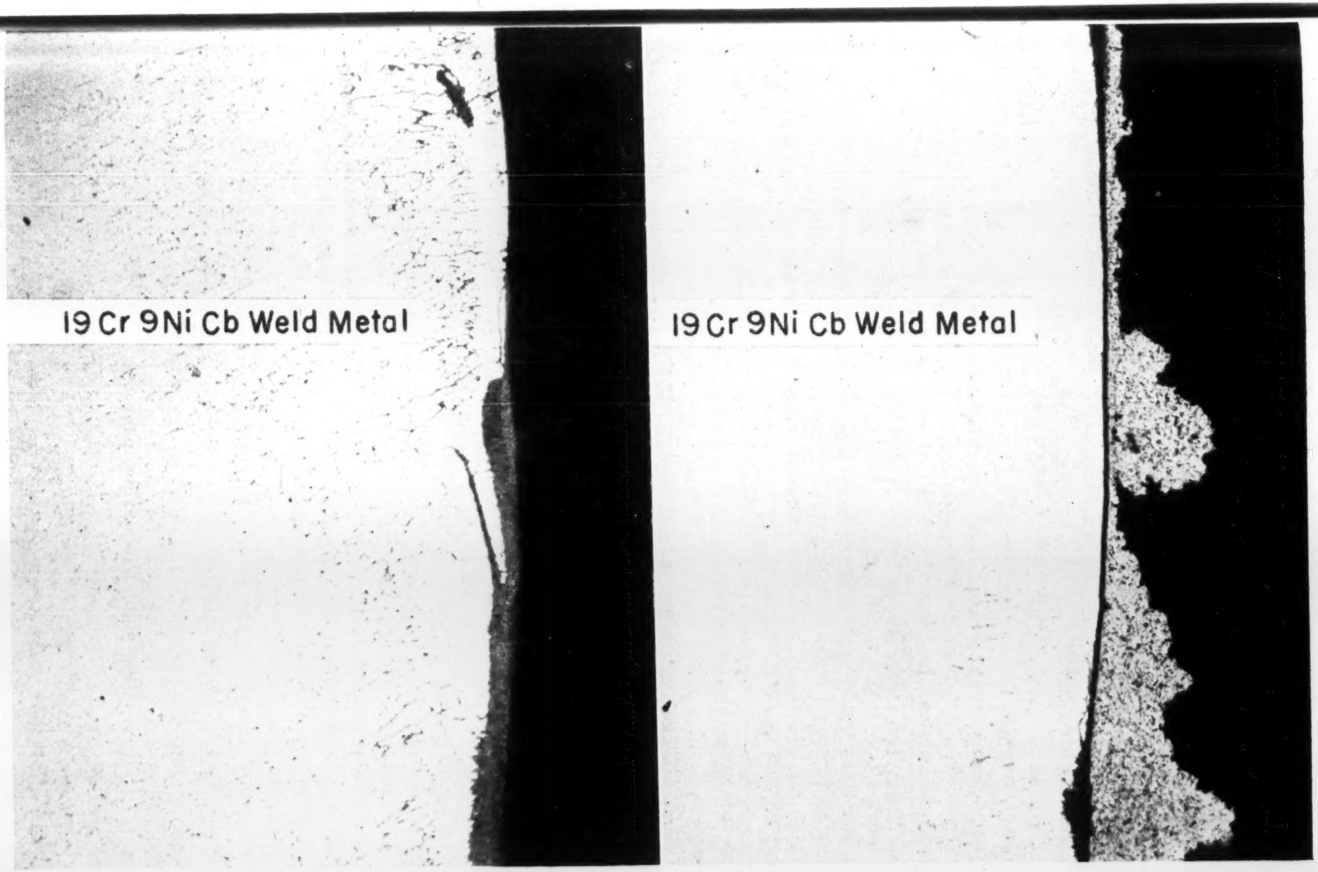


FIG. 18.—Microhardness Survey of Weld Junction.


19 Cr 9Ni Cb Weld Metal
19 Cr 9Ni Cb Weld Metal

Alkaline Chromate Etch (×100) Picral Etch (×100)

FIG. 19.—Path of Fracture in Room Temperature Transverse Weld Tension Specimen Removed from a Crack-Free Section of the 16Cr-13Ni-2Mo Cb to $2\frac{1}{4}$Cr-1Mo Weld Joint After Cycling Test.

Note that rupture occurred entirely in the $2\frac{1}{4}$ Cr-1 Mo metal adjacent to the fusion zone.

than the yield point stress at room temperature plus the limiting creep stress at operating temperature. Plastic deformation can therefore take place both at room temperature and at the operating temperature end of the cycle. The destructive action is aggravated by the fact that the two types of plastic deformation take place in opposite directions and undoubtedly occur in the weakest part of the joint, namely, in the narrow carbon depleted zone next to the weld junction. Stress concentration at the weld metal - pipe metal interface promotes the formation and inward propagation of an oxide notch in the less oxidation-resistant 2¼ Cr-1 Mo pipe surface, facilitates "intergranular oxygen infiltration," grain boundary oxidation, and subsequent cracking. The relative potency of the factors involved can not be evaluated without further experiments. Certain tests designed to shed light on this point are already under way.

TABLE III.—MECHANICAL WELD JOINT PROPERTIES.

	Yield Point, psi.		Tensile Strength, psi.		Elongation, per cent[a]	
TESTED AT ROOM TEMPERATURE						
Comparison weldment (as welded):						
Forging, 2¼ Cr-1 Mo	...		...		...	
Weld metal, 19 Cr-9 Ni Cb	...		...		...	
Forging, 18 Cr-8 Ni Cb	56 750		88 500		70.0	
	Ruptured in 18 Cr-8 Ni Cb forging which was unaffected by the weld					
16 Cr-13 Ni-2 Mo Cb to 2¼ Cr-1 Mo joint after cyclic heating test:	(1)	(2)	(1)	(2)	(1)	(2)
Pipe, 2¼ Cr-1 Mo	33 900	34 000	46 600	52 000	1.3	3.3
Weld metal, 19 Cr-9 Ni Cb	...	...	...	...	...	...
Casting, 16 Cr-13 Ni-2 Mo Cb	46 600	50 250	...	...	1.3	2.0
	Ruptured in 2¼ Cr-1 Mo pipe adjacent to weld metal					
TESTED AT 1100 F.						
16 Cr-13 Ni-2 Mo Cb to 2¼ Cr-1 Mo joint after cyclic heating test:						
Pipe, 2¼ Cr-1 Mo	20 500		26 300		2.0	
Weld metal, 19 Cr-9 Ni Cb	...		...		...	
Casting, 16 Cr-13 Ni-2 Mo Cb	...		...		...	
	Ruptured in 2¼ Cr-1 Mo pipe adjacent to weld metal					

[a] Elongation values were determined for a 1.5-in. gage length (from edge of weld metal to shoulder of specimen) for the 2¼ Cr-1 Mo section and for a ¾-in. gage length (from edge of weld metal to shoulder of specimen) for the 18 Cr-8 Ni Cb and 16 Cr-13 Ni-2 Mo Cb materials, respectively.

Acknowledgment:

The authors acknowledge the aid and advice received from L. F. Kooistra, Director of the Materials Research Laboratories of the Research and Development Dept. of The Babcock & Wilcox Co., Alliance, Ohio, and P. M. Brister, Staff Engineering Dept. of The Babcock & Wilcox Co., New York, N. Y.

[See Joint Discussion, page 858]

SOME CONSIDERATIONS IN THE JOINING OF DISSIMILAR METALS FOR HIGH-TEMPERATURE HIGH-PRESSURE SERVICE*

By O. R. Carpenter[1], N. C. Jessen[1], J. L. Oberg[1], and R. D. Wylie[1]

Synopsis

This paper relates to the metallurgical factors, the performance, and fabrication of dissimilar metal joints as may be employed in high-pressure, high-temperature steam service. It has been recognized by those who have considered or who have used this construction that expansion stresses existing between a ferritic material and an austenitic material might present some unknown problems in service. The expansion stress effects at dissimilar metal joints have led to the necessary consideration of other factors which were found to be present when a dissimilar metal weld is operating at high temperature, under high stresses in a cyclic manner. These are basically metallurgical ones. They are dependent upon the time, the temperature, and the combined stresses. Under some conditions, laboratory tests show that failure occurs in a carbon-depleted region of the heat-affected zone of the weld. Cyclic conditions, oxide penetration, and metallurgical or mechanical notches, as may occur under some operating or fabricating conditions, speed the time to failure. Failures of field welds of dissimilar metals under severe operating conditions are shown as examples.

This work does not predict service life expectancy of dissimilar metal joints. Its object has been, first, to point out the potentiality of this type failure in service and, second, to find remedies which remove or lessen the possibility of failures in dissimilar metal welds in high-temperature service. Work is, therefore, reported on welding techniques and developments designed to accomplish this end. Stabilization of carbides in the ferritic materials has been studied and is reported in this paper. Fabrication procedures best adapted to reduce failure are also discussed. Finally, test results are given to show that these considerations do much to reduce the potentiality of service failures of joints between ferritic and austenitic materials.

During the past several years, outlet steam temperature of central station boilers has been increased to 1050 F. at pressures of over 2000 psi. Higher temperatures might now be employed by designers if the metals-engineering were sufficiently advanced to permit satisfactory operation of metals in gas temperatures necessary to produce the required steam temperature. Advances in the use of metals for operation at high temperatures have been successfully made in the gas turbine industry. However, where boilers are concerned, the problem is more complex since not only must the metal successfully withstand oxidation, but it must also be suitable for the manufacture of tubular

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[1] Director, Welding Engineer, and Metallurgists, respectively, Works Control Laboratory, The Babcock & Wilcox Co., Barberton, Ohio.

materials, must carry internal pressure at the metal temperature, and, because of the large quantity of tubular material required, must be economical.

The load-carrying characteristics of metals at high temperatures, together with the economics involved, generally dictate to the boiler designer the use of both austenitic and ferritic materials for the outlet components of steam generating units. These components are the superheater and often the main steam line to the turbine. Several years ago when boiler units having outlet steam temperatures of 1050 F. were being designed, the Babcock & Wilcox Co. began a study of the problems concerned with the joining of austenitic to ferritic materials when these junctions would be operating under the pressure and temperature conditions required for making 1050 F. steam. The problem, in so far as the boiler unit design was concerned, related particularly to the superheater since these elements are located in gas temperatures ranging from 1900 to 2500 F.

This temperature range allows the successful use of ferritic materials for a large part of the superheater section, but requires austenitic materials at the outlet side. A study was therefore begun in 1947 of the dissimilar metal welds at the junction of austenitic to ferritic tubing at the metal temperatures to be expected when operating in the above gas temperatures. At the same time turbine and piping people were finding applications where dissimilar metal junctions were required. While the work reported in this paper deals only with tubing and weld metal, it is believed that the results may also be applicable where piping is involved.

The problem of joining austenitic to ferritic materials is not a particularly new one. There is considerable work reported in the literature. Recent work reported has given experience with piping. Of particular note is that dealing with the main steam lines of a 1050 F. boiler reported by H. Weisberg (**2**).[2] A paper was given by W. C. Stewart and W. G. Schreitz (**3**) dealing with thermal shock testing of austenitic and ferritic piping. Both of these papers describe transition type welds between dissimilar materials. Stewart reports a tendency of the 25 Cr-20 Ni[3] and the 19 Cr-9 Ni Cb welds, which joined austenitic to ferritic materials, to crack, the cracks appearing as fissures in the weld metal and revealed by oil-powder methods of inspection. No other tendency toward failure was noted after thermal shock testing.

The work reported here involves studies under accelerated conditions which are considerably more severe than are to be expected in service. In general, the test specimens were operated at service temperatures and pressures and with a superimposed mechanical or thermal shock added to the specimen. It had been our observation that while many reports of successful transition welds between dissimilar metals are available, none of these indicated service at the combined temperatures and pressures necessary for the high-temperature superheaters being considered. For example, in 1943 the Babcock & Wilcox Co. built a separately fired superheater which operated at a steam temperature of 1400 F. and at a pressure of 75 psi. This superheater contained transition welds between 18 Cr, 8 Ni Cb tubes and 9 Cr-1 Mo tubing. Those welds have operated satisfactorily. Earlier applications in boilers have been used but in all cases at lower steam temperatures and generally at lower pressures.

The object of this investigation has not been to predict a service-life expectancy of a weld between an austenitic and ferritic material operating at high temperatures and pressures. It has been to compare the high-temperature strength of austenitic and ferritic tubing with similar samples containing a butt weld joining austenitic to ferritic tubing. This comparison was made in a fatigue machine capable of testing full sections of superheater tubing when operating with internal pressure and at operating temperatures by cyclic quench equipment, and by means of stress-rupture testing.

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[2] The boldface numbers in parentheses refer to the references appended to this paper, see p. 49.

[3] 25 per cent chromium, 20 per cent nickel alloy steel; additional elements are indicated by the chemical symbols, Cb, Ti, etc.

Specifications

Capacity	60 000 in.-lb.
Speed	20 to 350 rpm.
Temperature, max.	1400 F
Pressure, max.	4000 psi.
Specimen Dia., max.	2.75 in.
Specimen Length	30 in.
Size of Motor	2 hp.

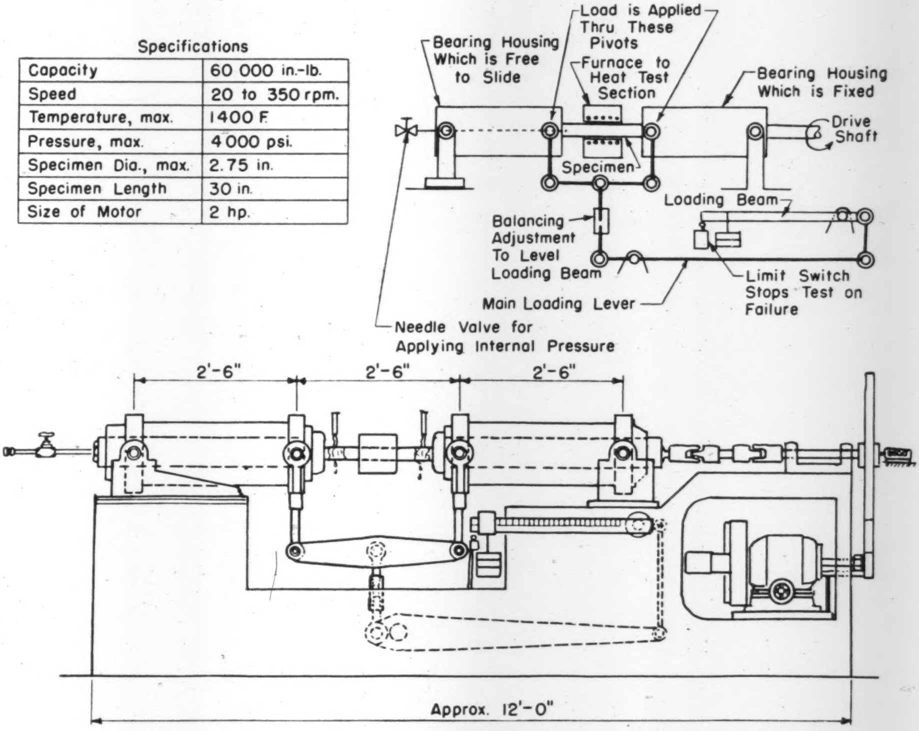

Load is Applied Thru These Pivots
Bearing Housing Which is Free to Slide
Furnace to Heat Test Section
Bearing Housing Which is Fixed
Drive Shaft
Specimen
Loading Beam
Balancing Adjustment To Level Loading Beam
Limit Switch Stops Test on Failure
Main Loading Lever
Needle Valve for Applying Internal Pressure
2'-6"
2'-6"
2'-6"
Approx. 12'-0"

FIG. 1.—Rotating-Beam Fatigue Machine for Testing Tube Weld Specimens.

These tests have not only demonstrated the effects of the differential expansion stresses but have shown additional factors which are significant in the life of joints forming transitions between dissimilar materials. This paper largely deals with a discussion of these and with certain procedures which may improve the life expectancy of the dissimilar metal joint.

The factors which have been found to influence the service of these welds are, aside from the expansion stresses, basically metallurgical ones, such as carbon depletion in the heat-affected zone of the ferritic material, oxide notches occurring in the ferritic material, and often in the carbon-depleted ferrite region, micro-fissuring of welds, and accelerated creep at the line of fusion.

Different weld metals are discussed and particularly the requirement of properly balancing the ferrite and austenite in 19 Cr-9 Ni welds in order to avoid fissuring. The strength of certain weld metals in stress-rupture is given, and finally certain modifications of technique are discussed which have been shown to increase the life under fatigue of transition welds between austenitic and ferritic materials.

FATIGUE TESTS

Description of Rotating Beam Fatigue Machine:

Figure 1 shows a diagrammatic sketch of the rotating-beam fatigue machine. This equipment accommodates tubular specimens up to 2.75 in. in diameter and 30 in. in length. Its general construction employs the features of a standard machine of this type with special provision for heating the specimen externally to any desired temperature and for maintaining an internal pressure at the inside of the specimen.

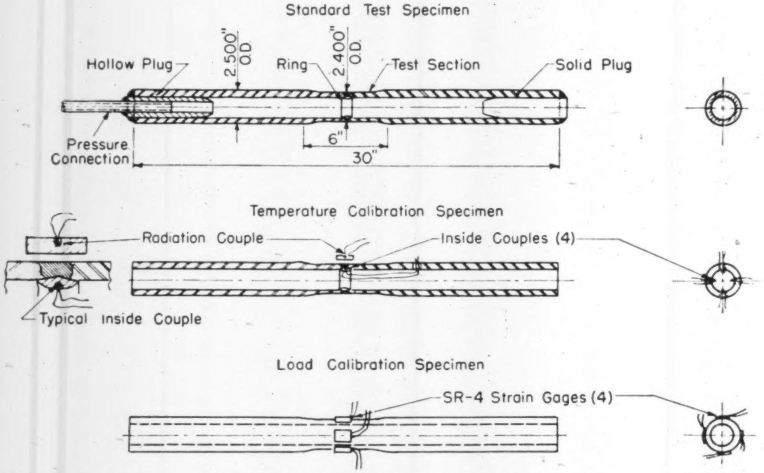

Standard Test Specimen
2.500" O.D.
2.400" O.D.
Hollow Plug
Ring
Test Section
Solid Plug
Pressure Connection
6"
30"
Temperature Calibration Specimen
Radiation Couple
Inside Couples (4)
Typical Inside Couple
Load Calibration Specimen
SR-4 Strain Gages (4)

FIG. 2.—Tube Weld Fatigue Test Specimens for Rotating Beam Fatigue Testing Machine.

The specimen is held between a driven shaft and an idling shaft by means of collets in such a manner that the assembly acts as a simple beam loaded at the one-third points. The idling assembly is arranged to slide, which eliminates all end-thrust due to bending. The load is applied to the specimen through a main loading lever and calibrated weighing arm. All bearings in the loading system are oilite coated with "Molykote" to reduce frictional errors.

The machine is driven by a 2-hp. motor coupled through a variable-speed drive so as to provide a speed range of 20 to 350 rpm.

The specimen is heated by a drum-type furnace at the point of maximum load. Temperatures are measured and controlled by means of a radiation-type thermocouple and a suitable temperature recorder. Automatic controls are arranged to shut down the machine in the event of failure of the specimen. Pressures up to 4000 psi. may be applied to the inside of the specimen by means of the hollow collet drawbar on the idling end of the machine. The pressurizing medium used is dry nitrogen. The number of cycles is measured by means of a reset counter on the driven shaft.

The tests described were made of tubing having an outside diameter of $2\frac{1}{2}$ in. and a wall thickness of $\frac{1}{2}$ in. In order to produce maximum fiber stress in the heated area, the specimens were machined to a reduced outside diameter of 2.40 in. as shown in Fig. 2. The ends of the reduced diameter were carefully faded into the original to avoid stress raisers.

The means of maintaining internal pressure is shown in Fig. 2. The pressure connection was brought out through the hollow drawbar for connection to the nitrogen source.

The calibration of the machine was accomplished by means of a standard specimen shown in Fig. 2. Four chromel-alumel thermocouples were peened into a backing ring and welded into the specimen shown. This permitted a study of the inside tube temperature with the outside, as measured by the radiation thermocouple. The specimen was then run in the machine at various temperatures indicated by the radiation thermocouple. These tests indicated that the radiation couple was accurate to within ±10 deg. Fahr.

TABLE I.—MATERIAL USED IN ROTATING BEAM FATIGUE TEST.

Material	Physical Form	Chemical Analysis, per cent								
		Carbon	Manganese	Silicon	Chromium	Nickel	Molybdenum	Columbium	Sulfur	Phosphorus
18Cr-8Ni Cb	Tubing[a]	0.08	1.52	0.31	18.55	11.36	0.06	0.86	0.016	0.013
Croloy 2¼	Tubing[a]	0.13	0.44	0.48	2.18	....	0.87		0.016	0.016
Croloy 5 Ti	Tubing[a]	0.07	0.49	0.31	4.82	....	0.50	0.05 Ti	0.016	0.016
25Cr-20Ni	Weld Metal	0.22	2.10	0.45	25.23	21.14	....	....	0.029	0.011
19Cr-9Ni Cb	Weld Metal	0.09	1.62	0.53	19.96	9.78	....	1.06	....	....
Croloy 2¼ low Carbon	Weld Metal	0.05	0.57	0.49	2.40	0.08	0.88	....	0.017	0.017

[a] 2½ in. outside diameter by 0.500 in. minimum wall.

A second specimen was made for calibrating the loading system of the machine. SR-4 type gages were attached to the specimen as shown. Gages were also attached to the arms of the loading yoke.

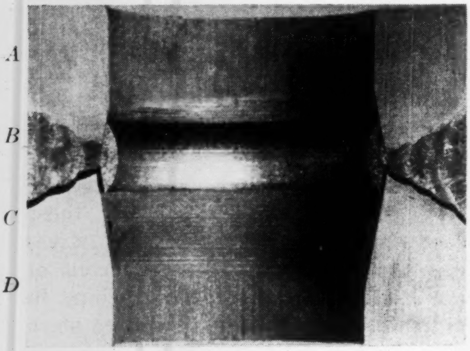
Stress readings were taken with the machine at rest and rotating as increasing loads were applied by means of the machine loading arm. In this manner, applied load was related to fiber stress in the specimen. Comparison of static loads to loads while the specimen rotated showed a loading accuracy of ±2 per cent.

Types of Tests and Materials Tested in Fatigue:

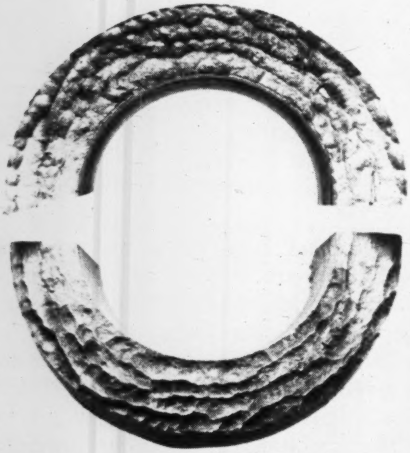
The fatigue tests described were run at temperatures and pressures which might be expected in the high-temperature section of a superheater of a boiler designed to operate with a steam outlet temperature of 1050 F. and a pressure of 2000 psi. In addition to these conditions, a superimposed fiber stress was applied to the joint by means of the loading system of the machine in order to accelerate failure and to show comparative *S-N* curves of varied predetermined conditions. After a number of tests had been made to learn the machine characteristics and to establish curves for unwelded tubing operating under similar loads, a number of materials and combinations of materials, as given in Table I, were tested. Fifty-nine fatigue tests have been run.

The preliminary tests on unwelded 18 Cr-8 Ni Cb (type 347) tubing established the type of specimen suitable for this investigation. Original tests on welds between 18 Cr-8 Ni Cb and 2¼ Cr-1 Mo tubing showed the importance of weld design upon fatigue life. These early results also established a typical failure pattern. The fracture was found to follow the line of fusion of the weld joining the austenitic to the ferritic tubing. A typical failure is shown in

Fig. 3. Subsequent tests were devoted to studies of the effect upon fatigue life and this type failure of welding techniques, heat treatments employed, composition of the base metal and weld metals.



A—18 Cr-8 Ni Cb
B—19 Cr-9 Ni Cb Weld
C—Location of Fracture Shown Below
D—$2\frac{1}{4}$ Cr-1 Mo



Fracture Surface at Point C

FIG. 3.—Typical Fatigue Fracture of an 18 Cr-8 Ni Cb-$2\frac{1}{4}$ Cr-1 Mo Tubular Fatigue Specimen.

Types of Weld Grooves Studied:

Welding technique was found to be effective in varying the fatigue life. Of particular note was the weld groove design. Figure 4 shows some of the weld grooves tested. It was found that any notches, as produced by backing rings or excess weld metal, may seriously impair the life of the weldment and, therefore, must be eliminated. The effect on fatigue life of the various designs shown in Fig. 4 is given in Table II. Different weld metals were tested. It was found from both fatigue and stress-rupture tests that in many of the respects being considered, 19 Cr-9 Ni Cb weld metal was superior to 25 Cr-20 Ni weld metal.

The Effect of Post Weld Heat Treatment:

After the first few dissimilar metal specimens failed and were examined metallographically, it was apparent that the phenomenon of carbon migration was one of the important factors in determining the mode of fracture. It was also clear that the use of a stress-relieving heat treatment at 1350 F., common practice for many ferritic steels, accelerated the migration of carbon and thus lowered the fatigue strength of the joint. Tests were made on dissimilar metal welds "as-welded" and after a heat treatment which was conducive to carbon migration. The strength decreased markedly after the heat treatment as shown in Table III.

These results indicate the severe effect of heat treatment on the fatigue strength of the dissimilar metal weld. It also shows that after 168 hr. at 1150 F. in the absence of applied stress, not enough migration has occurred to lower the strength of the weld appreciably. This substantiates the fact that stress accelerates carbon migration.

On the basis of these tests and on other stress-relieving tests, it was recommended that dissimilar metal welds be placed in service in the "as-welded" condition since any post heat treatment lowered strength of the weld, and since

TABLE II.—FATIGUE TEST DATA FOR SPECIMENS WITH DIFFERENT WELD DESIGNS.

Sample[a]	Heat Treatment[b]	Test Temperature, deg. Fahr.	Internal Pressure, psi.	Applied Stress, psi.	Cycles to Failure
3A	1350 F. for ½ hr., F.C.	1150	2000	17 310	89 573
3B	1350 F. for ½ hr., F.C.	1150	2000	16 800	409 032
3C	1350 F. for ½ hr., F.C.	1150	2000	16 900	116 469
3D	1350 F. for ½ hr., F.C.	1150	2000	16 700	568 097
3E	1350 F. for ½ hr., F.C.	1150	2000	17 200	213 300
3F	1350 F. for ½ hr., F.C.	1150	2000	17 320	240 361

[a] See Fig. 4 for description of weld design.

[b] F.C. = furnace cooled.

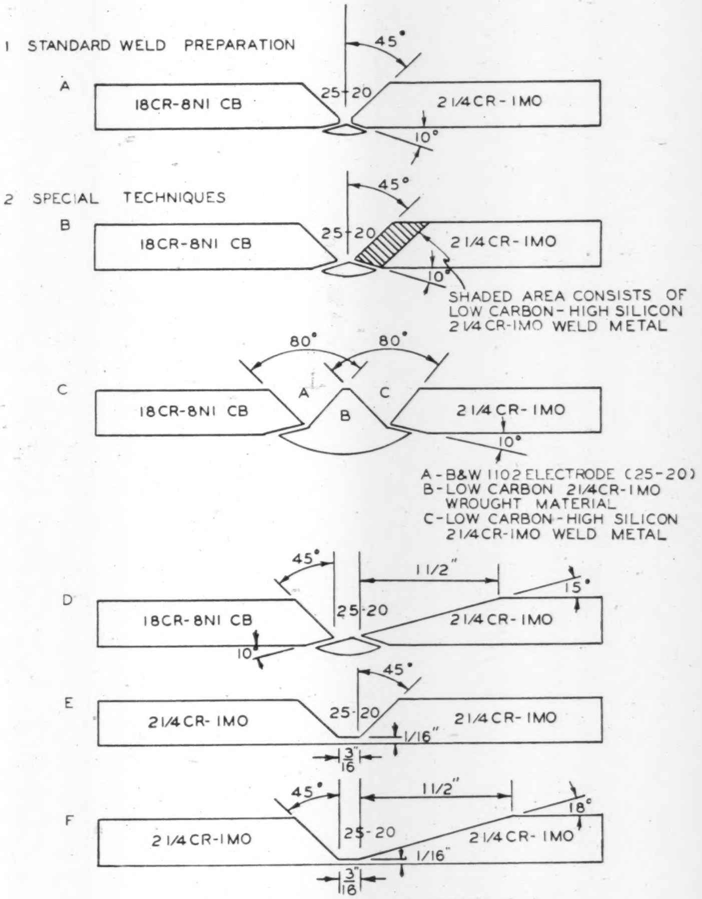

1 STANDARD WELD PREPARATION
A
18CR-8NI CB
25-20
21/4CR-1MO
45°
10°
2 SPECIAL TECHNIQUES
B
18CR-8NI CB
25-20
21/4CR-1MO
45°
10°
SHADED AREA CONSISTS OF LOW CARBON-HIGH SILICON 21/4CR-1MO WELD METAL
C
80°
80°
A
B
C
18CR-8NI CB
21/4CR-1MO
10°
A-B&W 1102 ELECTRODE (25-20)
B-LOW CARBON 21/4CR-1MO WROUGHT MATERIAL
C-LOW CARBON-HIGH SILICON 21/4CR-1MO WELD METAL
D
45°
1 1/2"
15°
18CR-8NI CB
25-20
21/4CR-1MO
10
E
45°
21/4CR-1MO
25-20
21/4CR-1MO
1/16"
3/16"
F
45°
1 1/2"
18°
21/4CR-1MO
25-20
21/4CR-1MO
1/16"
3/16"

FIG. 4.—Standard and Special Weld Techniques.

service temperatures would generally produce adequate softening of the welded zone.[4]

S-N Curves for Failure (Temperature and Pressure):

A number of standard fatigue specimens as shown in Fig. 5 were tested in the "as-welded" condition to develop stress *versus* cycles of stress reversal curves (commonly called *S-N* curves), for the failure of this dissimilar metal welded joint at 1050 F., and at 1150 F. Actually these points do not all fall on a curve but rather show a trend of results. For purposes of comparison, all tests were run with an internal pressure of 2000 psi. The curves for 1050 F. and 1150 F. are shown in Figs. 6 and 7.

TABLE III.—EFFECT OF PRIOR HEAT TREATMENT ON FATIGUE STRENGTH OF DISSIMILAR METAL WELD 18 CR-8 NI CB—2¼ CR- 1 MO WELDED WITH 19 CR-9 NI CB.

Heat Treatment	Test Temperature, deg. Fahr.	Stress, psi.	Cycles to Failure
As welded	1150	17 300	117 475
1350 F. for ½ hr., F.C.[a]	1150	17 310	89 573
1350 F. for 24 hr., F.C.[a]	1150	16 900	24 477
1150 F. for 168 hr., A.C.[b]	1150	17 420	109 000

[a] F.C. = Furnace cooled.
[b] A.C. = Air cooled.

It will be noted that the 1050 F. curve becomes asymptotic to the horizontal axis at approximately 16,000 psi. stress. The strength of the dissimilar metal weld is shown to be not greatly lower at 1050 F. than a weld between two 18 Cr-8 Ni Cb tubes operating at 1050 F.

Two types of failure occurred at 1050 F. Specimens with high external fiber stresses failed through the 2¼ Cr tube normal to the applied stress. The failure started at the junction of the austenitic weld and ferritic tube. Specimens run at lower stresses and for longer times failed along the line of fusion of the weld in the carbon depleted zone.

At 1150 F. the strength drops off and the endurance limit approaches 8000 psi. The failures followed the line of fusion of the welds in the decarburized area. At this temperature all of the points do not fall on a smooth curve, but rather show the general spread as might be expected at lower stresses.

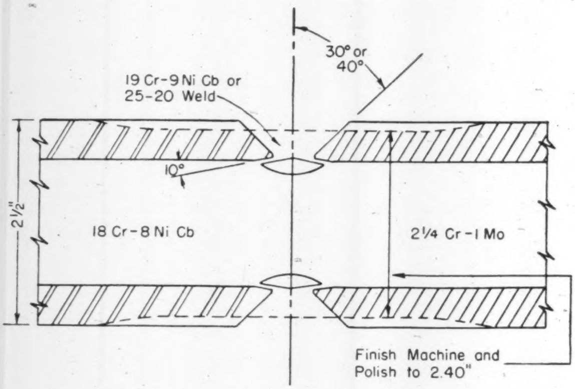

30° or 40°
19 Cr-9 Ni Cb or 25-20 Weld
10°
2½"
18 Cr-8 Ni Cb
2¼ Cr-1 Mo
Finish Machine and Polish to 2.40"

FIG. 5.—Standard Dimensions of Fatigue Specimen.

Since carbon migration was found to be an important factor in regulating fatigue strength, specimens were made joining 5 Cr-0.5 Mo Ti tubing to 18 Cr-8 Ni Cb tubing with 19 Cr-9 Ni Cb weld metal. The results of these fatigue tests are shown in Table IV. Failures in these specimens all occurred away from the line of fusion and after a heat treatment designed to produce carbon migration.

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[4] See Appendix.

These results indicate that the titanium-stabilized 5 Cr-0.5 Mo base materials will improve fatigue results. However, other factors may reduce the evaluation of these results, the most important of these being high-temperature oxidation fatigue and lower high-temperature strength.

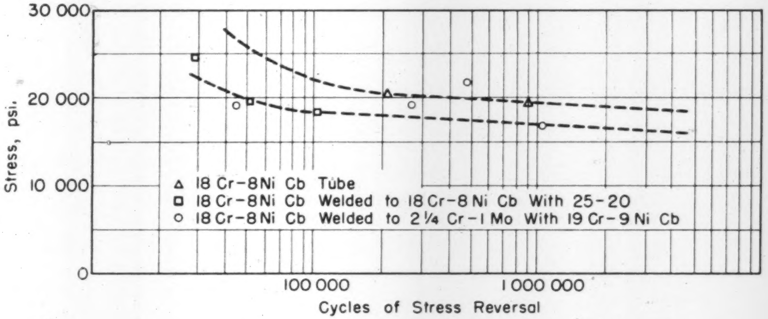


FIG. 6.—*S-N* Curve for Tubular Specimens at 1050 F.

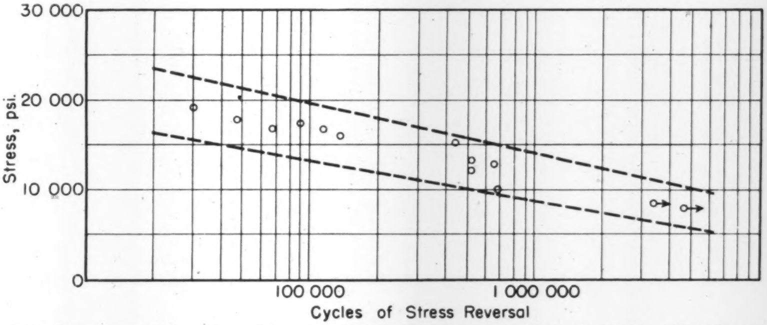


FIG. 7.—*S-N* Curve for Dissimilar Metal Tubular Specimen at 1150 F. 18 Cr-8 Ni Cb and $2\frac{1}{4}$ Cr-1 Mo Tubing Welding with 19 Cr-9 Ni Cb.

TABLE IV.—FATIGUE TEST DATA ON 5 CR-0.5 MO TI—18 CR-8 NI CB SPECIMENS COMPARED TO A STANDARD WELD SPECIMEN OF $2\frac{1}{4}$ CR-1 MO—18 CR-8 NI CB WELDED WITH 19 CR-9 NI CB.

Sample	Heat Treatment[a]	Test Temperature, deg. Fahr.	Internal Pressure, psi.	Applied Stress, psi.	Cycles to Failure
102	As welded	1150	2000	10 175	4 015 084[b]
103	1350 F. for 24 hr., F.C.	1150	2000	10 525	4 237 590[b]
104	1350 F. for 24 hr., F.C.	1150	2000	15 500	1 638 306
22 ($2\frac{1}{4}$Cr-1Mo to 18Cr-8Ni Cb)	1350 F. for $\frac{1}{2}$ hr., F.C.	1150	2000	15 400	437 906

[a] F.C. = Furnace cooled.
[b] Did not break.

Examination of Failed Fatigue Specimens:

After each specimen failed it was carefully examined visually for evidence of defects which may have affected the fatigue strength and was then sectioned in order that the surface of the fracture could be seen. On some specimens, the concentric rings such as are usually associated with fatigue failures could be seen. The point of initiation of the failure varied with different specimens. Some of the fractures started at the root of the weld while others appeared to start at the outer surface at the junction of the weld metal and the $2\frac{1}{4}$ Cr tubing. Figure 8 shows a typical fractured specimen. Notice the strain lines on the surface of the tube. When this piece was sectioned the fracture appeared as shown. The weld bead contour was outlined by the fracture, and there were concentric rings which indicated that this failure propagated from the inside. In certain specimens, microscopic slag inclusions or porosity were shown on the fractured surface to be the point at which the failure originated.

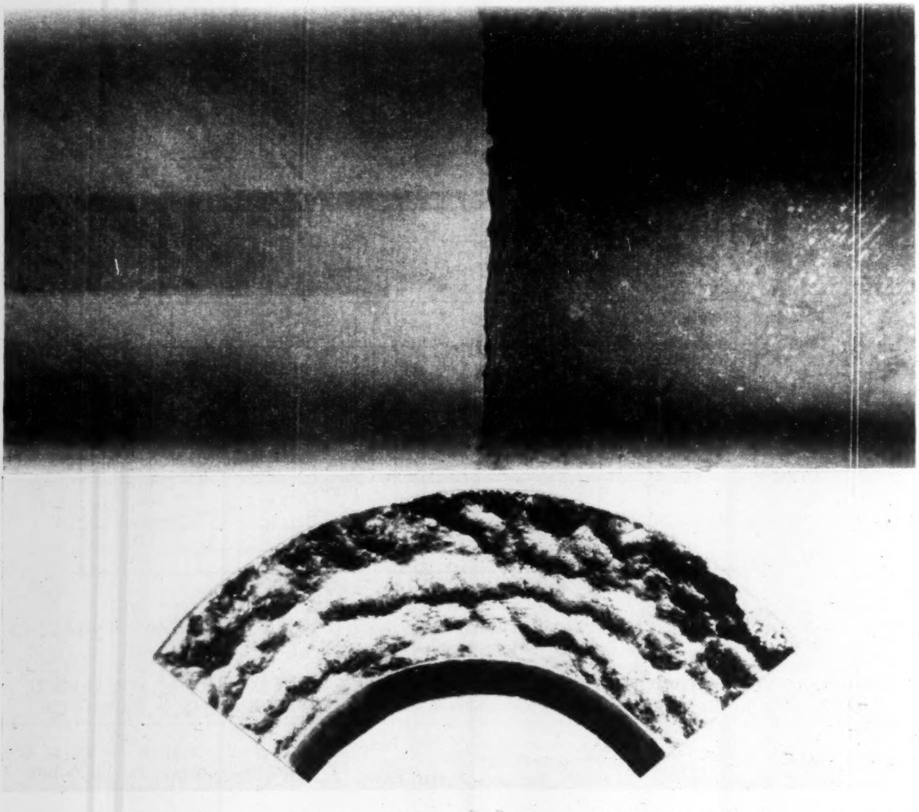


FIG. 8.—Specimen Surface and Fracture With the Concentric Circles Generally Representative of Fatigue Failure.

After a visual examination of the specimen, micro-examinations were made in order to reveal the mode and initiation point of the fracture. Certain additional and pertinent factors were observed. Of importance was the oxidation fatigue cracking adjacent to the weld in the ferritic tubing. All of the specimens examined showed carbon migration excepting those made up with titanium-stabilized tubing. The amount of carbon migration observed was found to be dependent upon the post-weld heat treatment, the length of time of the test, the test temperature, and the magnitude of the applied or combined stresses. Figure 9 illustrates typical cracks in the fatigue failures studied. Note the decarburization adjacent to the fusion line of the austenitic weld. Figure 10 illustrates oxidation fatigue cracking such as has been found at both the inside and outside surface of the specimens studied.

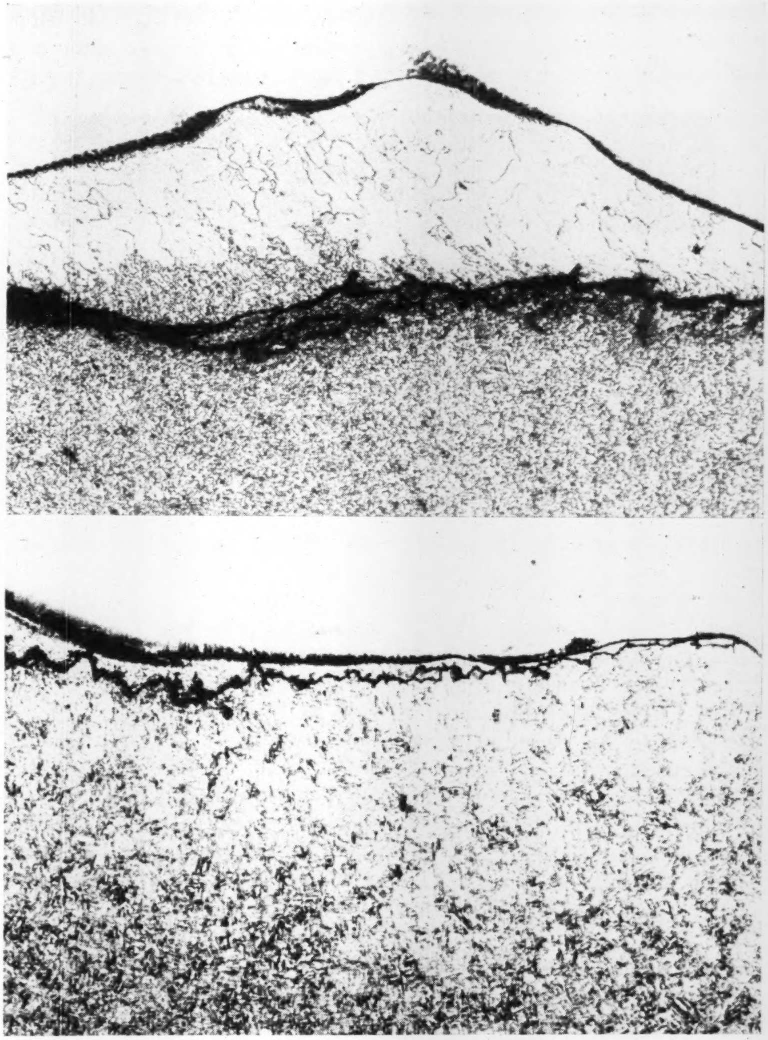


FIG. 9.—Typical Cracks Which Have Occurred in Fatigue Specimens (×100).

STUDIES OF DISSIMILAR METAL JOINTS NOT UNDER PRESSURE

STRESSES

In some boiler designs, horizontal superheater sections are supported from the boiler wall by means of austenitic castings which may be welded to ferritic superheater tubes by means of austenitic weld metals. These cast supports are usually unprotected from direct radiation from the gases in the boiler and are consequently often operating at high temperatures at the exposed areas. The edge welded to the ferritic tube is under restraint due to the lower tube temperature and its greater coefficient of expansion. These lugs must withstand severe service conditions such as temperature fluctuations and possible vibrating forces. In general, the loads carried are conservative. Experience has shown, however, that under some conditions these lugs fail at the line of fusion at the tube. A short discussion of the mode of these failures is of interest.

Several examples of superheater support lug failures are shown in Fig. 11. Notice that the bead sequence is clearly seen and the failures resemble those produced by fatigue testing. Figure 12 shows an incipient failure of a superheater lug which has had 8 years service, and it also illustrates the oxidation fatigue effect due to the expansion stresses on the surface of the tube adjacent to the weld.

The examination of these lug welds illustrates exactly the same microstructural characteristics as shown in fatigue failures. The cracking usually followed the line of fusion of the weld through the low-carbon zone, and apparently was initiated at either the external junction of weld and tube or at the non-fused zone at the base of the lug.

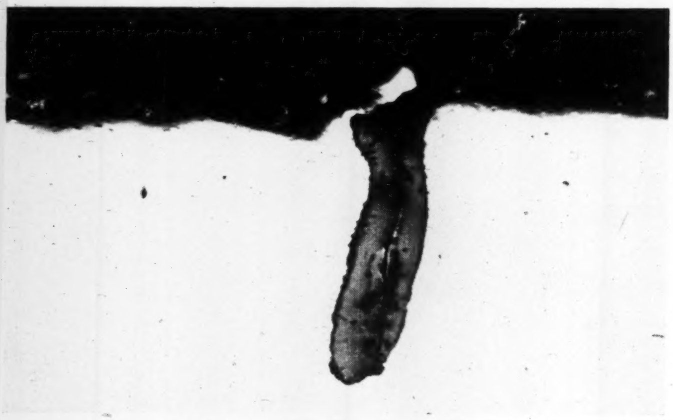


FIG. 10.—Oxidation Fatigue Crack on Fatigue Specimen Adjacent to Weld (×250).

Oxidation fatigue cracking shown at higher magnification in Fig. 13 is of the same type as shown in laboratory fatigue tests.

Although carbon migration is not too prominent, it may be observed in the failure shown in Fig. 14. This would possibly indicate that after a number of years of operation the carbon content of the decarburized zone may be replaced by diffusion from the unaffected tube material.

Laboratory Reproduction of Superheater Lug Failures:

Specimens were prepared with standard 25 Cr-12 Ni cast lugs welded on a $2\frac{1}{4}$ Cr-1 Mo tube with 25 Cr-20 Ni electrode in one case and a special low-carbon $2\frac{1}{4}$ Cr-1 Mo electrode in the second. After about 15 quenches from 1150 F. to 80 F., the lugs welded with 25 Cr-20 Ni separated from the tube, whereas those welded with the low-carbon $2\frac{1}{4}$ Cr-1 Mo electrode showed no visible cracking even after 100 quenches. These specimens are shown in Figs. 15 and 16.

Another similar test with titanium-stabilized 5 Cr-0.5 Mo as the tube material selected because of its resistance to carbide migration was run. A control sample was also included with a $2\frac{1}{4}$ Cr-1 Mo tube. Austenitic 25 Cr-12 Ni lugs were welded to the test tube with 25 Cr-20 Ni. After 50 quenches from 1150 F., the lug was partially separated from the $2\frac{1}{4}$ Cr-1 Mo tube, but there was no evidence of failure of the lugs on the titanium-stabilized 5 Cr-0.5 Mo tube. This substantiates the results of previous mechanical fatigue tests showing that in the absence of carbon migration there would be less likelihood of a failure in a dissimilar metal weld, other factors such as oxidation fatigue and high-temperature strength being neglected.

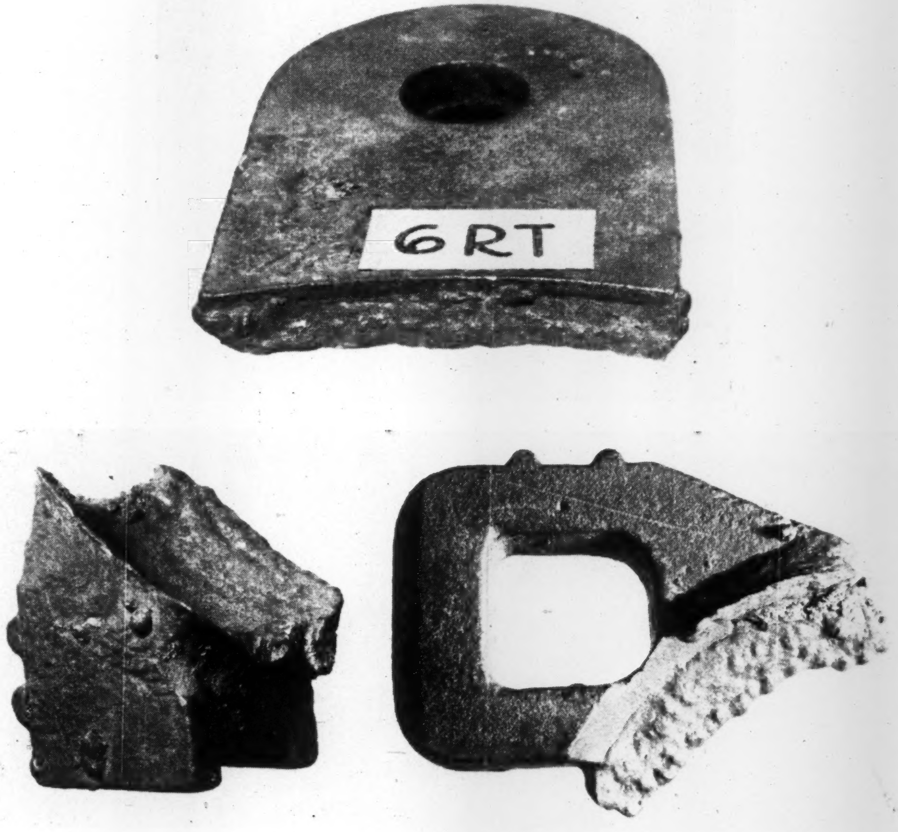
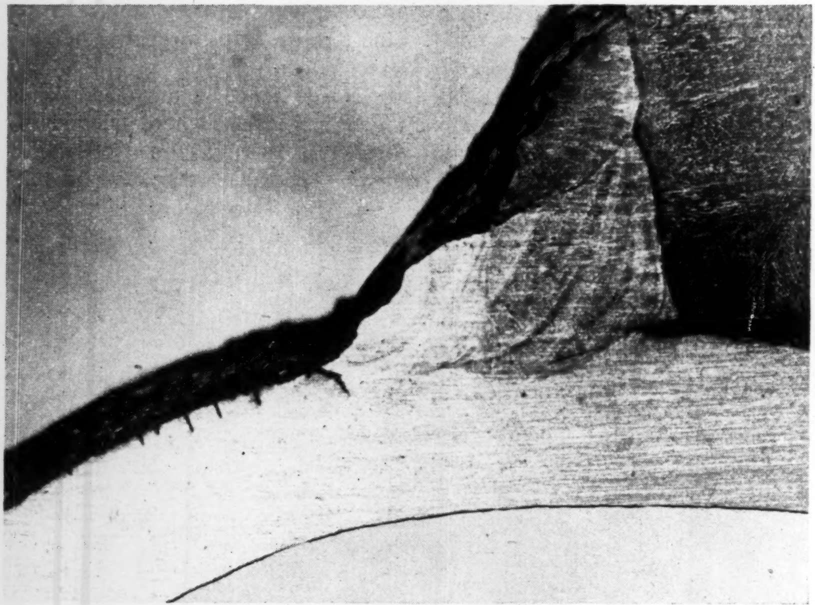


FIG. 11.—Examples of Superheater Support Lugs Which Have Failed in Service.

Another series of lug tests was made under conditions where quenching was done from the inside of a tubular specimen. The specimen was heated externally and quenched from the inside. It is shown in Fig. 17. One-half of the lugs were attached to the tube with 25 Cr-20 Ni weld metal, the other half with special low-carbon $2\frac{1}{4}$ Cr-1 Mo. After 388 cycles from 1120 F. (weld temperature) none of the welds was visibly affected. However, microscopic


(×4)

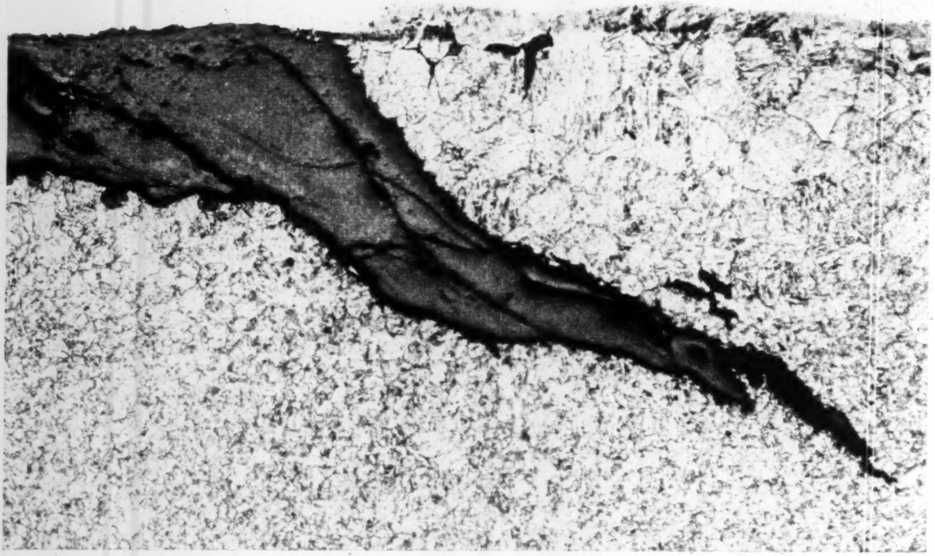

(×100)

FIG. 12.—Oxidation Fatigue Type Cracking Which Occurred Adjacent to a Support Lug After About 8 Years Service.

examination revealed that the welds prepared with 25 Cr-20 Ni electrodes were cracked about half way off the tube, and those welded with low carbon $2\frac{1}{4}$ Cr-1 Mo showed no signs of cracking along the line of fusion.

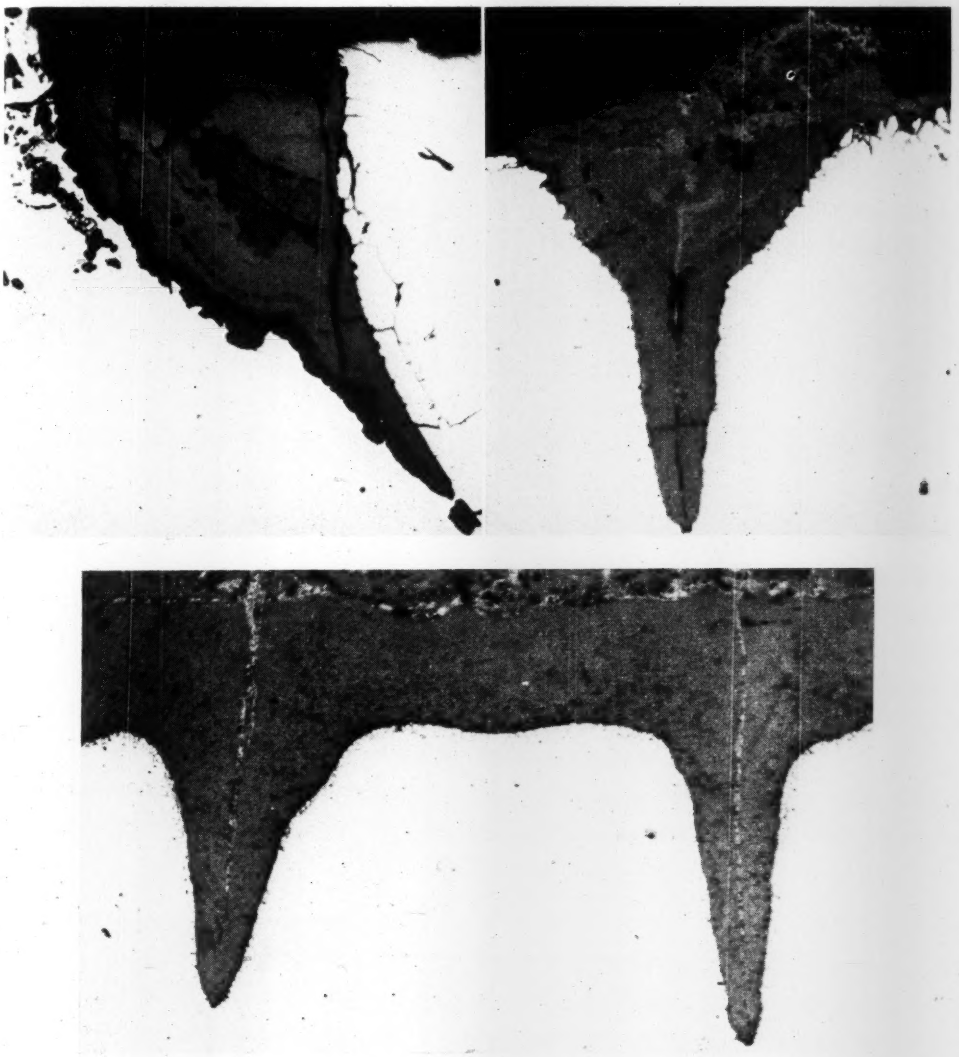


Fig. 13.—Examples of Accelerated Oxidation Adjacent to Austenitic Welds (×250).

The low-carbon chromium weld retards carbon migration. It reduces the difference in the thermal expansion coefficients normally present at the junction of the ferritic tube and an austenitic weld because of the dilution in the weld deposit. Figure 18 shows reduced carbon migration and no evidence of accelerated oxidation at the surface of the weld. This latter observation brings out the effect of expansion stresses in accelerating oxidation fatigue cracking.

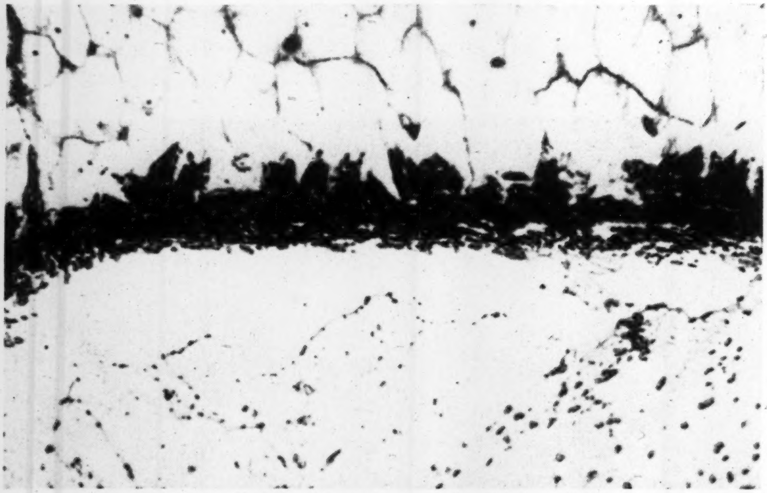


FIG. 14.—Carbon Migration in a Service Lug Failure.

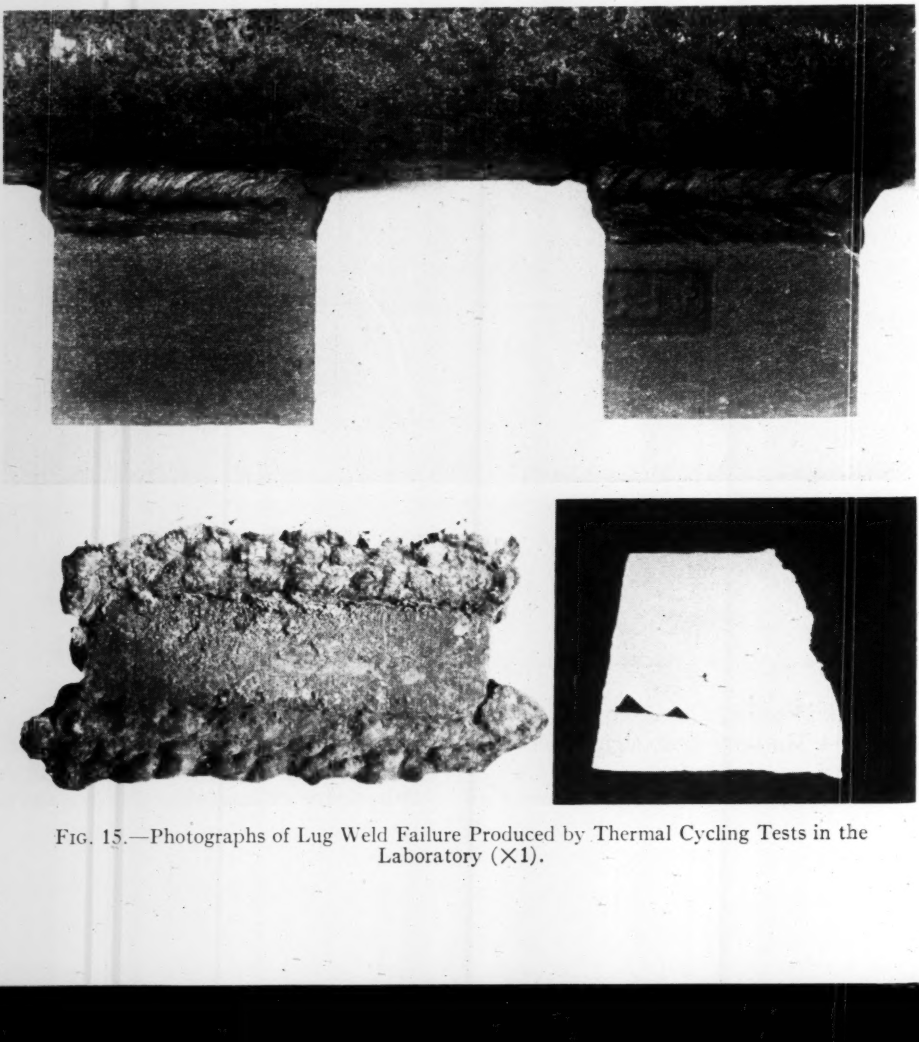


FIG. 15.—Photographs of Lug Weld Failure Produced by Thermal Cycling Tests in the Laboratory (×1).

CYCLIC QUENCH UNDER PRESSURE OF TUBULAR SPECIMENS

To study further the action in fatigue of dissimilar metal joints, a cyclic quench apparatus was built, shown schematically in Fig. 19.

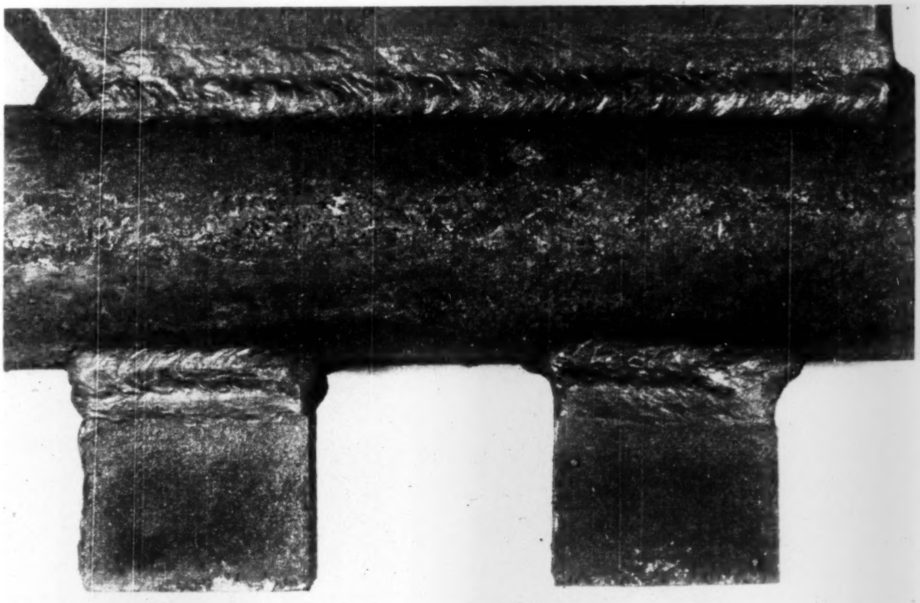
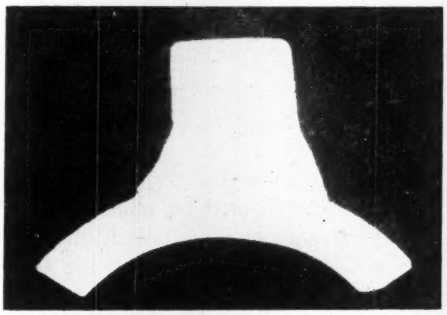


FIG. 16.—Excellent Results on Lug Specimen Welded with Low Carbon $2\frac{1}{4}$ Cr-1 Mo Electrode After Identical Thermal Cycling Tests (×1).



The test specimen is mounted in a gas-fired furnace with thermocouples welded to the outer surface of the tube to measure skin temperatures. A probe is inserted inside the tube to measure steam temperature. The cycle is controlled by an electronic timer and is fully automatic providing the following sequence of operations:

1. Fill the system with water.
2. Close both inlet and outlet valves.
3. Heat until the desired temperature is reached and the pressure regulating valve opens.
4. Bleed steam through the pressure regulating valve until the desired steam temperature is obtained.
5. Open inlet valve and quench system with high-pressure water from the hydraulic intensifier.
6. Open both valves and continue quench cycle to reduce specimen temperature to desired limit.
7. Close both valves and repeat (3), (4), (5), (6), and (7).

The quenching cycle may be initiated

by the skin temperature in the tube or the steam pressure, depending on the setting of the recording controllers. The duration of quench may be regulated by resetting the timer.

In order to protect the operator from the possibility of explosion of the heated tube, the furnace is built of $\frac{1}{2}$-in. thick steel plate, and the entire unit is operated behind a sheet steel screen. An emergency release is also provided to release pressure in the event that the timer fails to function.

The specimen may consist of a single piece of tubing or it may be made up of tubing containing a circumferential butt weld. The specimens discussed in this investigation were made of 18 Cr-8 Ni Cb tubing welded to $2\frac{1}{4}$ Cr tubing using the same sizes and welding details described for the fatigue tests. Attachment to the exterior piping was made by ring welding.

A number of specimens have been tested in this equipment, the results being given in Table V. The metal temperatures employed approximated that which might be expected in superheater application. The degree of quench is severe, but it will be observed that quenching always occurs with the system under pressure and that the temperature drop on the outside wall in the pressure quench portion of the cycle is not great.

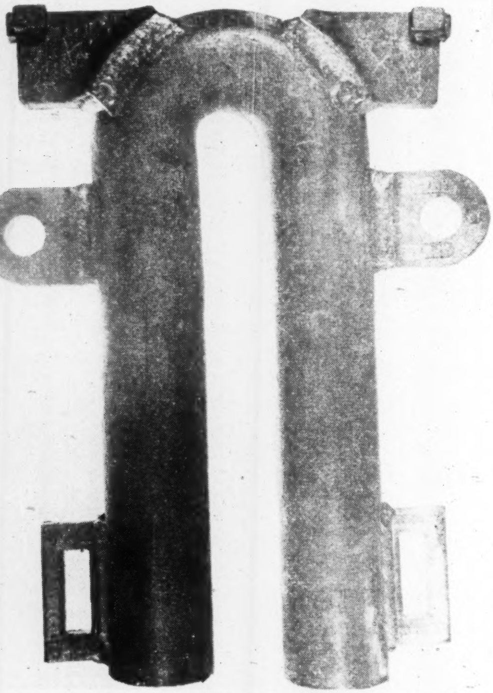


FIG. 17.—Specimen Design Used in Thermal Cycling Tests.

After control of the equipment was established, specimens ran for 107 to 318 cycles before a crack was observed. The cracks generally followed the line of fusion of the ferritic side of the weld.

In addition, this type of test produced corrosion-fatigue cracking on the inside of the specimens. Figure 20 shows a fusion zone crack from one pressure quench specimen. In these specimens which were tested in an as-welded condition little carbon migration was observed as a result of the short time at temperature. It indicates that the differential expansion stresses will produce failure without the presence of carbon migration, especially where notches are present to start a failure.

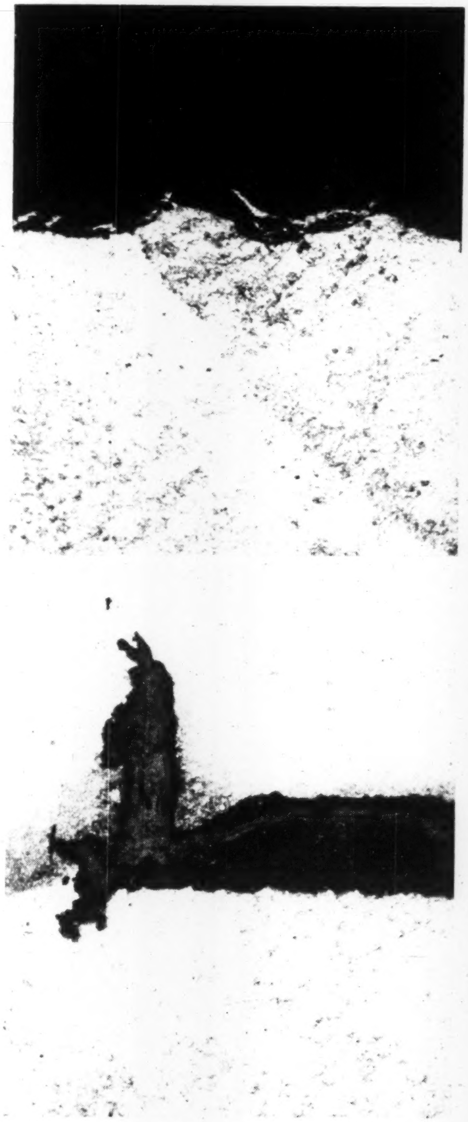


FIG. 18.—Some Carbon Migration at Junction Between Low Carbon 2¼ Cr Weld and 2¼ Cr Tube (×100).

Note that Surface Does Not Show Incipient Cracking

WELD METALS

The quality of the weld metal has been found to bear considerable influence upon the time of failure, under the conditions described, of dissimilar metal joints. The program of the Babcock & Wilcox Co. designed to study weld metal for this application is of interest and its findings bear importance in extending the life of dissimilar welds by reducing fissuring, microscopic inclusions, and cracking.

Austenitic weld metal has generally been the choice for welding dissimilar joints since the high tensile strength and ductility of the joints as welded makes them highly suitable. In addition, with proper welding procedure, undesirable dilution can be eliminated and weldments produced which are highly ductile when tested at room temperature. When operating at high temperature, however, because of expansion stresses of austenitic welds in this application, other properties such as stress-rupture, creep, thermal and mechanical fatigue properties and oxidation prove to be the criteria upon which serviceability of the joint is to be evaluated.

In the study of weld metal properties with particular reference to dissimilar metal joints, it was realized that the use of ferritic weld metal might be a sound procedure if dilution and cracking problems could be overcome. Consequently, studies reported here will give considerations concerning the effects of variations of composition on the properties of austenitic weld metals and test results and compositions of suitable ferritic weld metals.

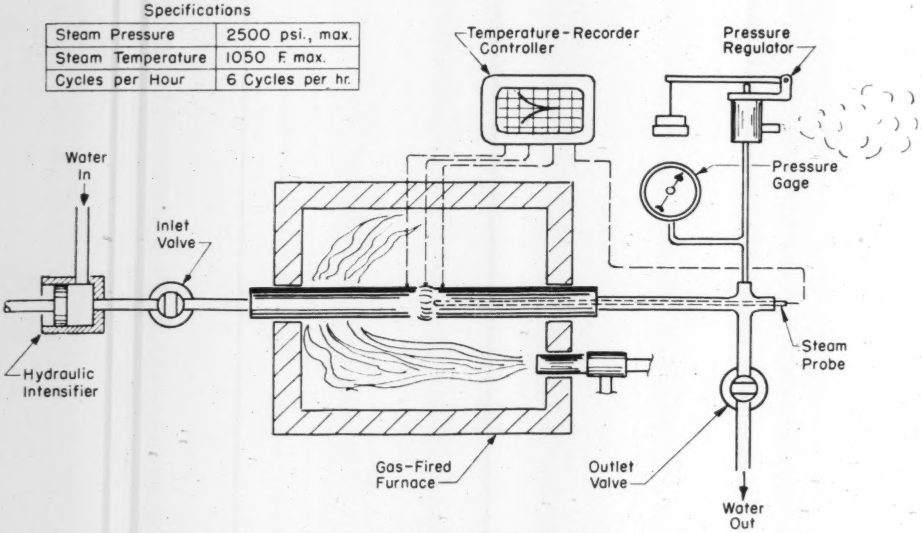

Specifications
Steam Pressure
2500 psi., max.
Steam Temperature
1050 F max.
Cycles per Hour
6 Cycles per hr.
Temperature-Recorder Controller
Pressure Regulator
Water In
Pressure Gage
Inlet Valve
Hydraulic Intensifier
Steam Probe
Gas-Fired Furnace
Outlet Valve
Water Out

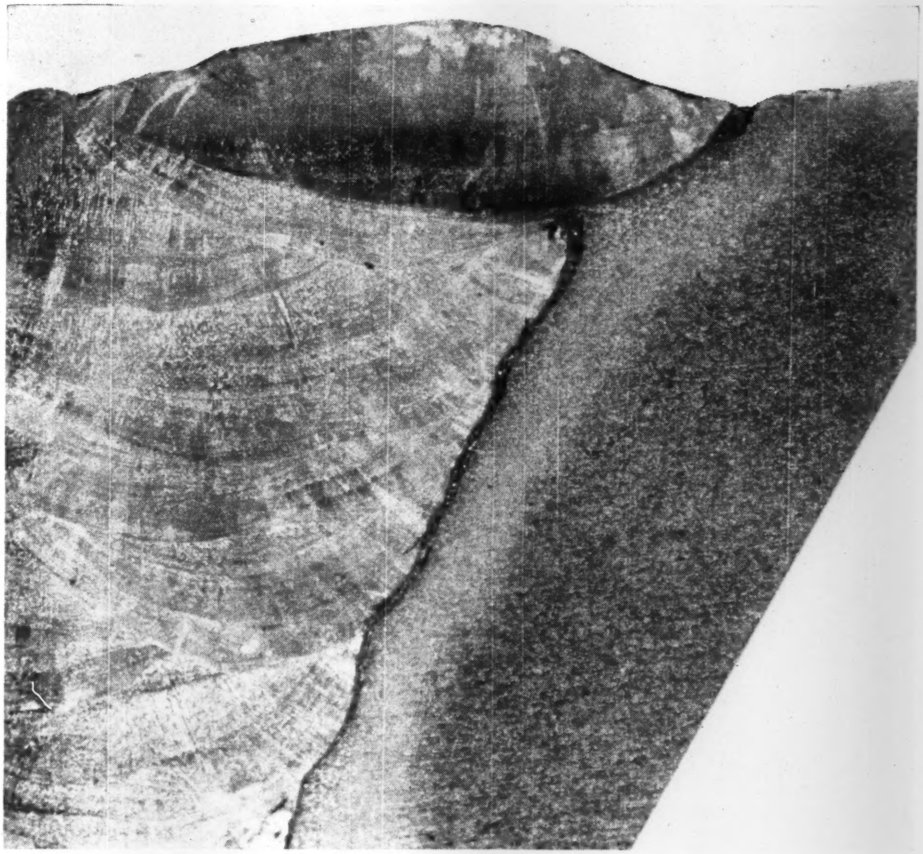
FIG. 19.—Cyclic Quench Apparatus for Testing Tube Weld Specimens at Operating Pressure and Temperature.

TABLE V.—CYCLIC QUENCH TESTS UNDER PRESSURE.

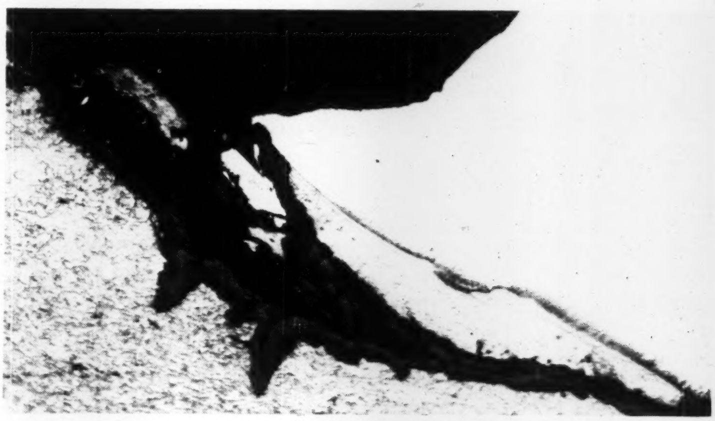
Specimen	Material	Steam Temperature, deg. Fahr.	Approximate Tube Temperature, deg. Fahr.	Pressure, psi.	Temperature Cycle, deg. Fahr.	Number of Cycles
No. 1	2¼ Cr-1 Mo to 18 Cr-8 Ni Cb Welded with 19 Cr-9 Ni Cb Electrode	850	1190	2000	1190-500	318
No. 2	2¼ Cr-1 Mo to 18 Cr-8 Ni Cb Welded with 19 Cr-9 Ni Cb Electrode	1050	1200	2300	1200-400	182
No. 3	2¼ Cr-1 Mo to 18 Cr-8 Ni Cb Welded with 19 Cr-9 Ni Cb Electrode	1050	1110	2300	1110-400	206
No. 4	5 Cr-½ Mo Ti to 18 Cr-8 Ni Cb Welded with 19 Cr-9 Ni Cb Electrode	1050	1300	2300	1300-480	107
No. 5	2¼ Cr-1 Mo to 18 Cr-8 Ni Cb Welded with 19 Cr-9 Ni Cb Electrode	760 1050	1050 1300	2300 2750	....	100 76
No. 6	2¼ Cr-1 Mo to 18 Cr-8 Ni Cb Welded with 19 Cr-9 Ni Cb Electrode	720 1050	1050 1280	2300 2750	....	100 81

Austenitic Weld Metals:

The large majority of welding of dissimilar materials with austenitic electrodes has been done in the past with 25 Cr-20 Ni electrodes. Metallurgical considerations which govern the quality of 25 Cr-20 Ni weld metal have been discussed in the literature (4) (5), and are today rather well established. The maintenance of a proper carbon-silicon ratio, in conjunction with low sulfur and phosphorus values has proven an adequate assurance of sound weld metal

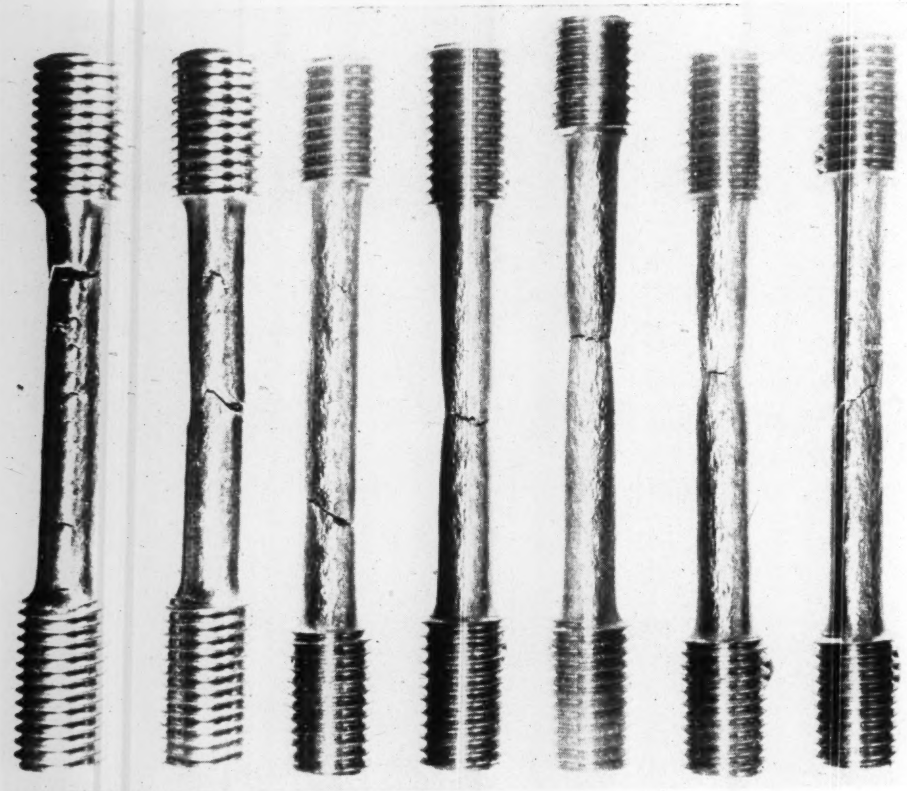


(*a*) Macro-Section (×10).



(*b*) Showing Initiation of Crack at Higher Magnification (×100).

FIG. 20.—Crack in First Cyclic Quench Specimen Showing 19 Cr-9 Ni Cb-$2\frac{1}{4}$ Cr Junction.



C.........	0.09	0.09	0.10	0.17	0.18	0.17	0.18
Si........	0.35	0.38	0.85	0.41	0.48	0.62	0.70
Specimen	E-121	E-122	E-645	E-646	E-647	E-648	E-649

FIG. 21.—Tension Specimens Showing Effects of Carbon and Silicon Content on Ductility of Totally Austentic 19 Cr-9 Ni Weld Deposit.

Note Absence of Fissures in Higher Carbon Alloys.

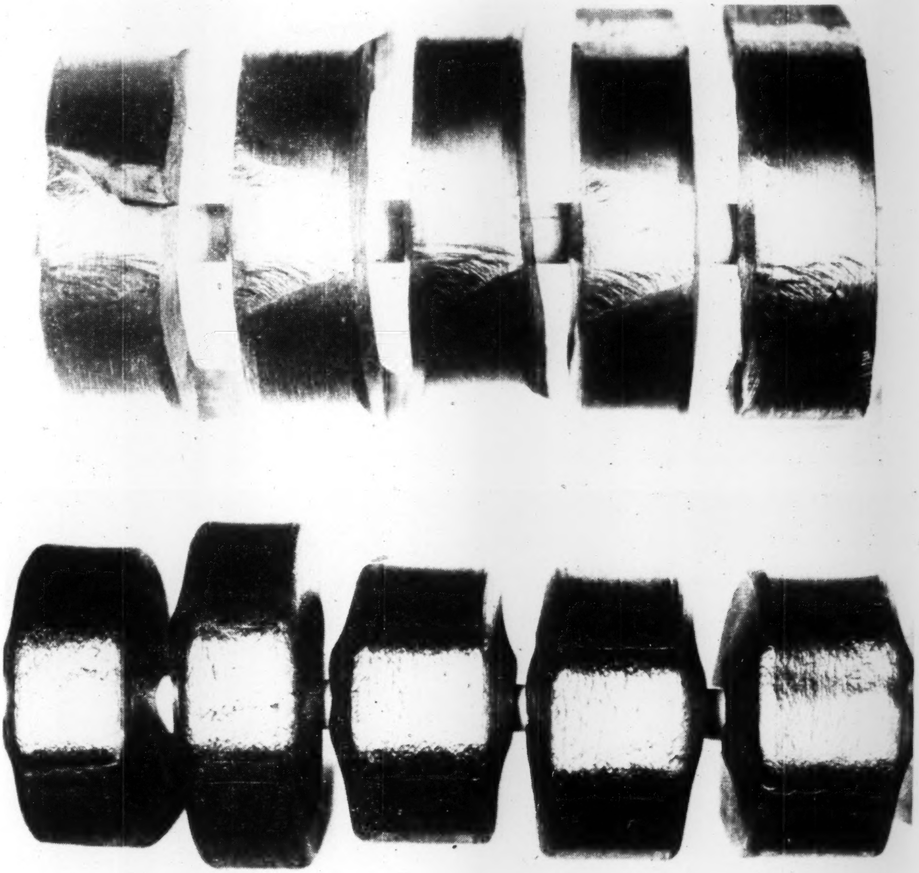
TABLE VI.—EFFECTS OF CARBON AND SILICON ON THE PROPERTIES OF FULLY AUSTENITIC 19 CR-9 NI WELD METALS IN THE "AS-WELDED" CONDITION.

Specimen	Yield Point, psi.	Tensile Strength, psi.	Elongation, per cent	Reduction of Area, per cent	Fissures	Chemical Analysis, per cent							Microstructure[a]
						C	Mn	Si	Cr	Ni	S	P	
E-121...	54 000	70 750	13.0	27.8	badly fissured	0.09	2.19	0.35	17.74	13.99	0.015	0.015	fully austenitic
E-122...	57 500	79 000	28.5	44.5	slightly fissured	0.09	2.27	0.38	18.30	11.64	0.017	0.011	fully austenitic
E-654...	38 250	58 640	23.0	44.7	very badly fissured	0.07	1.92	0.75	18.03	12.64	0.010	0.012	fully austenitic
E-645...	35 500	73 000	36.5	42.8	badly fissured	0.10	2.05	0.85	17.97	11.96	0.012	0.014	fully austenitic
E-646...	45 500	91 000	43.0	57.0	none	0.17	2.04	0.41	18.43	8.92	0.012	0.012	fully austenitic
E-647...	51 500	85 250	53.0	62.2	none	0.18	2.02	0.48	18.74	9.12	0.016	0.012	fully austenitic
E-648...	49 000	80 000	47.0	64.4	very slight	0.17	2.14	0.62	18.63	9.48	0.015	0.012	fully austenitic
E-649...	48 000	79 000	41.5	39.8	slight	0.18	2.06	0.70	18.21	9.40	0.012	0.016	fully austenitic

[a] As determined from A. L. Schaeffler's constitution diagram.

quality. Since the 25 Cr-20 Ni composition is not considered as satisfactory for high-temperature high-stress applications, as the 19 Cr-9 Ni Cb type of analysis, the variables which affect the weld metal properties in the 19 Cr-9 Ni type of alloys were investigated.

The logical approach to this problem appeared to be a study of the fully austenitic 19 Cr-9 Ni type of weld deposits with varying carbon and silicon contents. Table VI is a presentation of some of the test data of fully austenitic 19 Cr-9 Ni weld metals. At carbon values of 0.10 per cent or below and silicon contents as low as 0.35 per cent, the mechanical properties of the weld metal are poor. Figures 21 and 22 show the tensile and bend specimens of the weld metals listed in Table VI. As was the case in the investigation of the 25 Cr-20 Ni alloys, fissuring results



C	0.10	0.17	0.18	0.17	0.18
Si	0.85	0.41	0.48	0.62	0.70
Specimen	E-645	E-646	E-647	E-648	E-649

Fig. 22.—Side (top) and Face (bottom) Bends of Weld Metals With Varying Carbon and Silicon Contents.

from the presence of non-metallic intergranular films of the type shown at a magnification of 1000 in Fig. 23. Increase in carbon content at silicon values below 0.50 per cent completely restores the ductility as indicated by the data obtained from specimens shown in Fig. 21. Increase in silicon content above about 0.50 per cent results in the reappearance of fissures even at the higher carbon level.

FIG. 23.—Intergranular Silicate Films Responsible for the Fissures Present in Low Carbon Austenitic 19 Cr-9 Ni Weld Deposits (×1000).

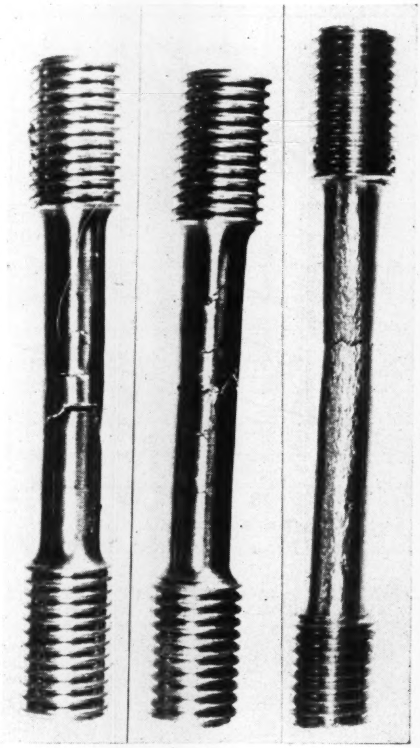
TABLE VII.—EFFECTS OF CARBON AND SILICON ON THE MECHANICAL PROPERTIES OF FULLY AUSTENITIC 19 CR-9 NI CB WELD METALS IN THE "AS-WELDED" CONDITION.

Specimen	Yield Point, psi.	Tensile Strength, psi.	Elongation, per cent	Reduction of Area, per cent	Fissures	Chemical Analysis, per cent								Microstructure[a]
						C	Mn	Si	Cr	Ni	Cb	S	P	
E-124...	48 500	48 500	7.0	8.9	badly fissured	0.10	2.20	0.39	17.60	14.84	0.96	0.017	0.014	fully austenitic
E-125...	51 500	58 500	7.0	13.0	badly fissured	0.09	1.93	0.39	17.88	13.54	0.73	0.020	0.013	fully austenitic
E-650...	60 620	92 980	36.0	52.3	none	0.18	2.00	0.70	18.21	9.40	1.25	0.010	0.011	fully austenitic

[a] As determined from A. L. Schaeffler's constitution diagram.

The addition of columbium to the 19 Cr-9 Ni weld metals of fully austenitic composition does not substantially affect the soundness. Table VII lists the physical properties and chemical analysis of one high-carbon and two low-carbon specimens of this analysis. Again severe fissuring with resulting low tensile strength and elongation occurs at the low carbon level although the silicon content was held at the low value of

0.39 per cent. Increase in carbon content to 0.18 per cent completely eliminates all evidence of fissuring and substantially improves both the tensile strength and elongation, even though the silicon content was at a slightly high value of 0.70 per cent. Figures 24 and 25 show the tensile and bend specimens of this series.

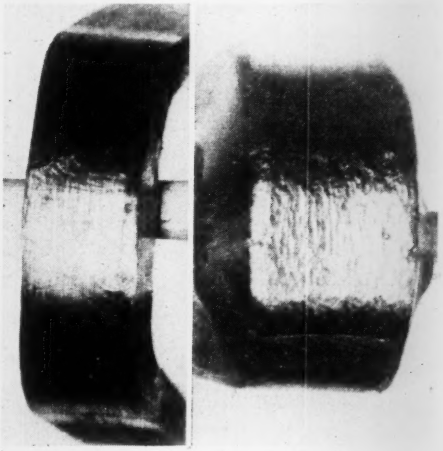


C	0.09	0.10	0.18
Si	0.39	0.39	0.70
Cb	0.73	0.96	1.25
Specimen	E-125	E-124	E-650

FIG. 24.—Effect of Carbon and Silicon on the Ductility of Austenitic 19 Cr-9 Ni Cb Weld Metal.

From the preceding data it is evident that sound, fully austenitic weld metals cannot be produced readily at carbon levels below 0.10 per cent. A decrease in silicon to near 0 per cent may possibly result in the elimination of fissures even at these low carbon values; however, the difficulties of producing weld metals at such low silicon levels would render this solution impractical.

It has been known for some time that the introduction of a small percentage of delta iron or ferrite into the microstructure of the low carbon 19 Cr-9 Ni type of weld metals affords an effective control of weld metal quality. The presence of ferrite can be readily determined and its percentage estimated by etching the polished specimen in Kallings reagent. For control purposes the quantity of ferrite present in the microstructure can be computed on the basis of a modified Maurer diagram proposed by A. L. Schaeffler. This diagram is shown in Fig. 26. After computing the chromium and nickel equivalents of the analysis the microstructural composition may be read directly.



Side Bend Face Bend

C	0.18
Si	0.70
Cb	1.25

FIG. 25.—Side and Face Bend Specimens of High Carbon-High Silicon Totally Austenitic 19 Cr-9 Ni Cb Weld Metal E-650.

A series of tests was, therefore, conducted with 19 Cr-9 Ni type weld metals containing about 4 to 12 per cent of delta iron or ferrite in the microstructure at varying carbon and silicon contents. Table VIII gives the mechanical properties of six 19 Cr-9 Ni weld metals at 0.08 to 0.10 per cent carbon and silicon contents from 0.42 to 0.92 per cent. No fissuring was observed in any of the specimens, and the tensile strength and elongation properties of these weld metals do not appear to be affected by the variation in silicon content. The 0.505 in. tension specimens and bend tests of this series are shown in Figs. 27 and 28, respectively.

A similar set of test specimens of columbium stabilized 19 Cr.-9 Ni weld metal is shown in Table IX. No fissuring was noted on any of the 21 tension and bend specimens regardless of silicon variations of from 0.31 to 1.01 per cent and carbon variation of 0.07 to 0.19 per cent. Tension and bend specimens are again shown in Figs. 29 and 30, respectively. Both carbon and columbium appear to increase the tensile strength of the weld metal. Increasing carbon content, with the ranges covered in this investigation and in the absence of columbium does not appear to reduce

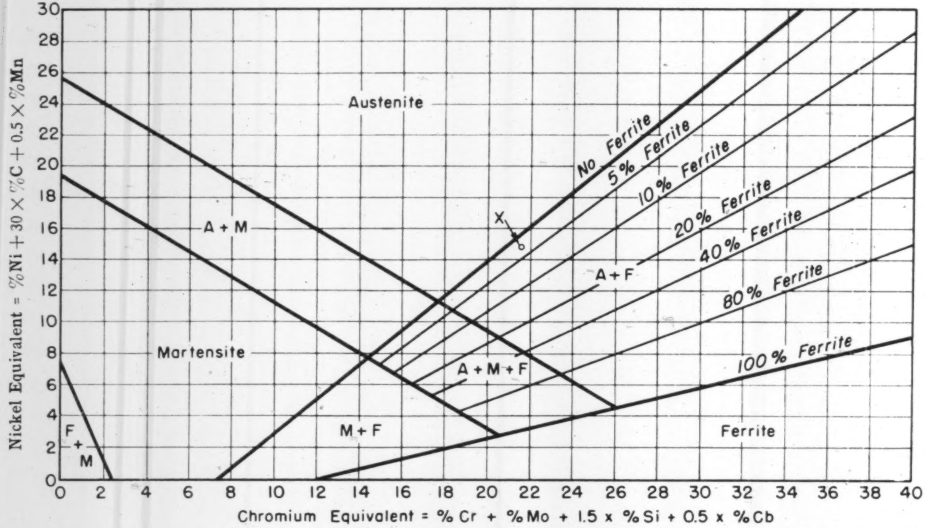


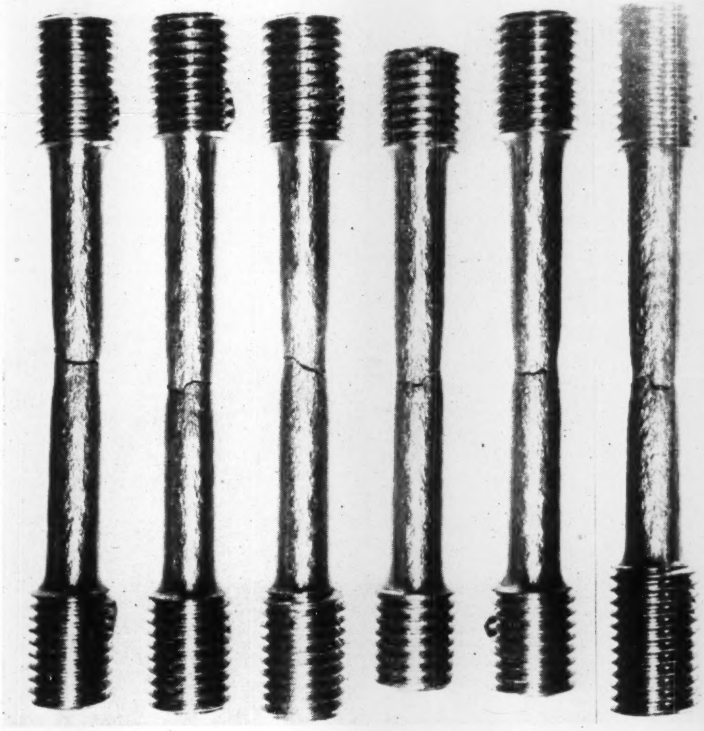
FIG. 26.—Constitution Diagram of Iron-Chromium-Nickel Weld Metals (*By Anton L. Schaeffler*(3)).

TABLE VIII.—EFFECTS OF CARBON AND SILICON CONTENT ON THE PROPERTIES OF PARTIALLY FERRITIC 19 CR-9 NI WELDS IN THE "AS-WELDED" CONDITION.

Specimen	Yield Point, psi.	Tensile Strength, psi.	Elongation, per cent	Reduction of Area, per cent	Fissures	Chemical Analysis, per cent							Microstructure, per cent Ferrite[a]
						C	Mn	Si	Cr	Ni	S	P	
E-624	53 240	84 040	44.5	59.5	none	0.10	2.00	0.42	19.21	9.08	0.010	0.018	3
E-625	50 520	84 180	47.0	56.5	none	0.09	2.00	0.53	19.13	9.00	0.012	0.010	5
E-626	51 340	85 560	46.5	56.7	none	0.10	2.02	0.68	19.10	9.16	0.007	0.013	4
E-627	49 920	86 090	46.5	65.7	none	0.10	2.10	0.82	19.13	8.92	0.007	0.011	5
E-628	50 530	86 900	46.5	68.5	none	0.10	1.96	0.89	18.85	8.60	0.012	0.010	6
E-653	48 000	81 000	49.0	64.7	none	0.08	2.04	0.92	18.81	9.48	0.013	0.013	4

[a] As determined from A. L. Schaeffler's constitution diagram (Fig. 26).

the ductility of the weld metal. Specimen E-647 at 0.18 per cent carbon and a fully austenitic structure showed 53 per cent elongation as compared to the partially ferritic specimen E-625 at 0.09 per cent carbon which showed 47 per cent elongation. Unfortunately, there was no high-carbon 19 Cr-9 Ni specimen showing a partially ferritic structure available for a more direct comparison.



C	0.10	0.09	0.10	0.10	0.10	0.08
Si	0.42	0.53	0.68	0.82	0.89	0.92
Specimen	E-624	E-625	E-626	E-627	E-628	E-653

FIG. 27.—Tension Specimens Showing Excellent Ductility of Partially Ferritic 19 Cr-9 Ni Weld Deposits.

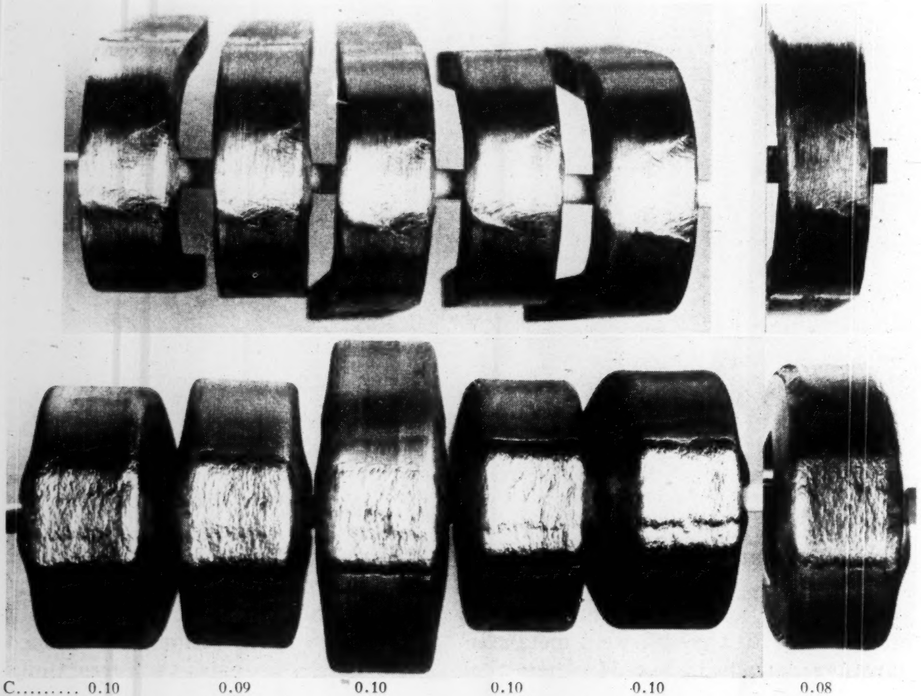
The effect of columbium on the properties of 19 Cr-9 Ni weld metals is shown graphically in Fig. 31, where the mechanical properties of partially ferritic 19 Cr-9 Ni weld metals containing columbium are plotted against the columbium content. Increasing columbium content results in increased tensility and decreased ductility of the weld metal.

Aside from the effect of columbium on the tensile properties of the weld deposit, the presence of columbium in a 19 Cr-9 Ni type weld metal results in the appearance of crater cracks. The cause, mode of formation, or for that matter any of the factors which influence the formation of these crater cracks, have not as yet been determined. These cracks are always intergranular and appear to result from the presence of an intergranular constituent in the microstructure, which does not, however, appear to be a slag type of constituent.

TABLE IX.—EFFECT OF CARBON AND SILICON CONTENTS ON THE PHYSICAL PROPERTIES OF PARTIALLY FERRITIC 19 CR-9 NI CB WELD METAL IN THE "AS-WELDED" CONDITION.

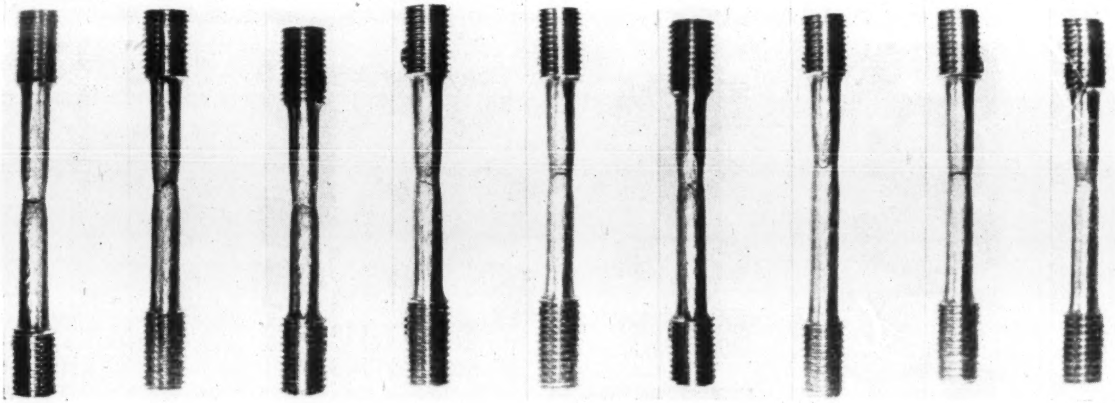
Specimen	Yield Point, psi.	Tensile Strength, psi.	Elongation, per cent	Reduction of Area, per cent	Fissures	Chemical Analysis, per cent								Microstructure, per cent Ferrite[a]
						C	Mn	Si	Cr	Ni	Cb	S	P	
E-102	64 000	91 500	41.0	58.1	none	0.08	2.01	0.36	18.87	8.68	0.35			5
E-103	62 000	92 750	40.0	50.0	none	0.08	2.20	0.31	19.15	9.21	0.71			3
E-104	68 000	95 500	30.5	55.7	none	0.08	2.18	0.41	19.04	9.25	1.12			5
E-105	68 500	96 000	32.5	48.6	none	0.08	2.27	0.52	18.83	9.02	1.61			6
E-655	52 850	86 560	45.0	59.9	none	0.07	1.94	0.94	18.74	9.60	0.39			5
E-656	55 600	88 500	42.0	52.8	none	0.07	1.94	1.00	18.40	9.68	0.79			5
E-618	61 000	89 500	41.0	61.0	none	0.10	2.04	0.42	18.85	9.04	0.44	0.013	0.017	8
E-619	56 500	89 750	42.5	62.1	none	0.11	1.98	0.54	19.03	9.00	0.43	0.017	0.013	2
E-620	61 650	91 720	42.5	57.6	none	0.12	2.24	0.65	18.85	8.84	0.39	0.012	0.014	3
E-621	56 870	89 830	42.5	57.0	none	0.11	2.04	0.76	18.78	8.80	0.39	0.016	0.017	4
E-657	58 000	93 000	37.5	58.3	none	0.10	2.10	0.72	18.46	8.64	0.85	0.020	0.016	5
E-658	62 900	92 860	36.0	60.5	none	0.10	2.07	0.95	18.40	8.62	0.72	0.013	0.020	5
E-622	58 380	90 340	40.5	57.8	none	0.10	2.06	0.84	18.74	8.88	0.41	0.020	0.011	5
E-641	63 000	94 000	37.5	59.8	none	0.10	2.02	0.78	18.00	8.52	1.00	0.009	0.014	3
E-642	61 000	94 250	36.5	51.8	none	0.10	2.05	0.86	18.37	8.56	1.40	0.010	0.013	5
E-643	61 400	96 120	37.0	56.7	none	0.10	2.00	0.96	18.29	8.48	1.02	0.012	0.012	5
E-644	52 500	95 000	38.5	52.8	none	0.10	2.03	1.01	18.29	8.46	1.39	0.017	0.014	6
E-408	69 500	100 625	37.8	48.0	none	0.16	1.99	0.52	18.73	8.84	0.82			5
E-410	68 500	97 875	37.3	52.1	none	0.14	1.73	0.62	19.40	9.48	0.65			5
E-651	65 200	101 100	34.0	45.7	none	0.17	2.04	0.69	18.43	8.40	1.39	0.009	0.011	9
E-652	66 700	102 600	33.5	45.1	none	0.19	2.06	0.97	18.91	8.40	1.47	0.017	0.017	3

[a] As determined from A. L. Schaeffler's constitution diagram (Fig. 26).

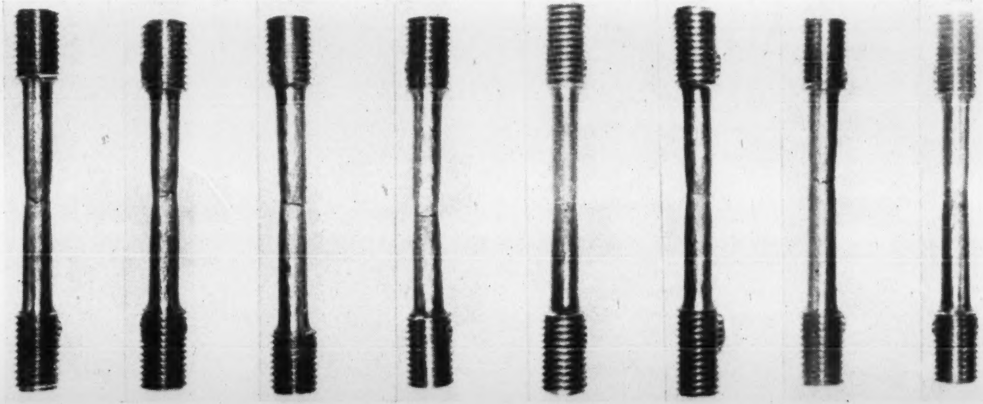


C	0.10	0.09	0.10	0.10	0.10	0.08
Si	0.42	0.53	0.68	0.82	0.89	0.92
Specimen	E-624	E-625	E-626	E-627	E-628	E-653

FIG. 28.—Side Bend (top) and Face Bend (bottom) Specimens of Partially Ferritic 19 Cr-9 Ni Weld Metal.



C........	0.07	0.07	0.10	0.10	0.10	0.10	0.10	0.10	0.10
Si........	0.94	1.00	0.42	0.72	0.95	0.84	0.78	0.86	0.96
Cb........	0.39	0.79	0.44	0.85	0.72	0.41	1.00	1.40	1.02
Specimen.	E-655	E-656	E-618	E-656	E-658	E-622	E-641	E-642	E-643



C........	0.10	0.11	0.11	0.12	0.16	0.14	0.17	0.19
Si........	1.01	0.54	0.76	0.65	0.52	0.62	0.69	0.97
Cb........	1.39	0.43	0.39	0.39	0.82	0.65	1.39	1.47
Specimen.	E-644	E-619	E-621	E-620	E-408	E-410	E-651	E-652

FIG. 29.—Tension Specimens Showing Excellent Ductility of Partially Ferritic 19 Cr-9 Ni Cb Weld Metals Regardless of Carbon, Silicon, or Columbium Contents.

Cracks appear regardless of whether the columbium is present in the base metal, the electrode, or both, and their size seems to be directly proportional to the amount of columbium present.

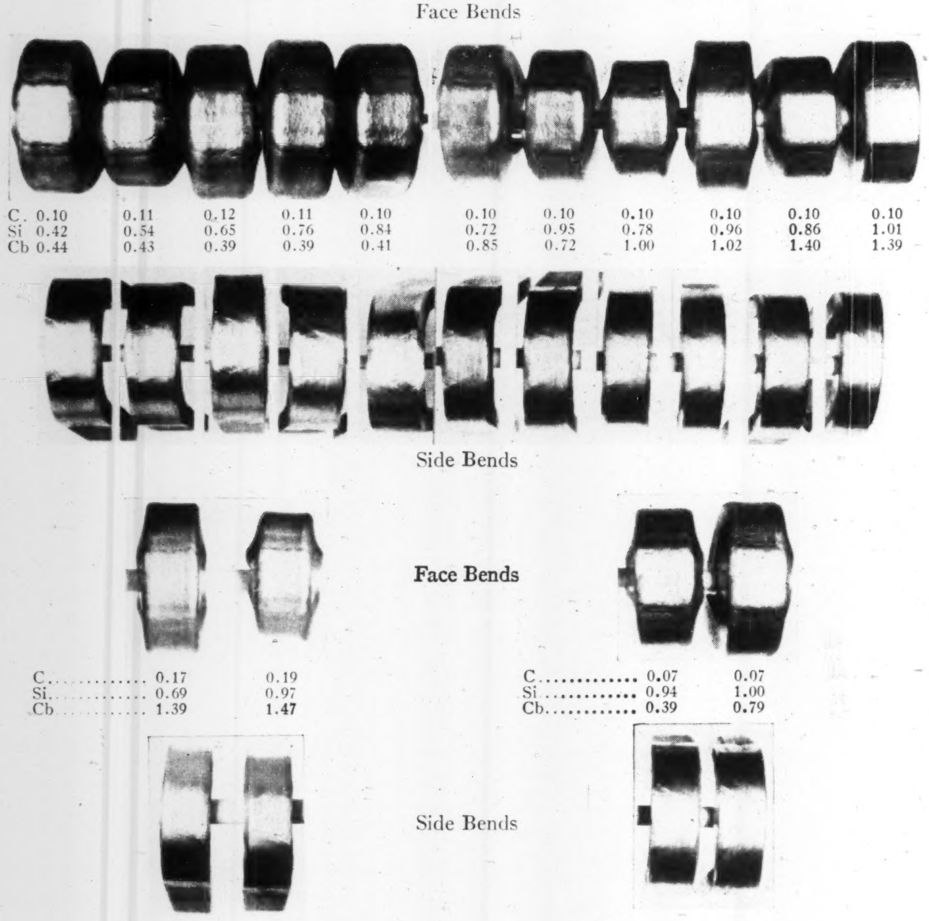

Face Bends
C. 0.10 0.11 0.12 0.11 0.10 0.10 0.10 0.10 0.10 0.10 0.10
Si 0.42 0.54 0.65 0.76 0.84 0.72 0.95 0.78 0.96 0.86 1.01
Cb 0.44 0.43 0.39 0.39 0.41 0.85 0.72 1.00 1.02 1.40 1.39
Side Bends
Face Bends
C............ 0.17 0.19
Si............ 0.69 0.97
Cb........... 1.39 1.47
C............ 0.07 0.07
Si............ 0.94 1.00
Cb........... 0.39 0.79
Side Bends

FIG. 30.—Bend Specimens Welded with Partially Ferritic 19 Cr-9 Ni Cb with Various Combinations of Carbon, Silicon, and Columbium.

Figure 32 shows the types of crater cracks which appear when columbium is present in the weld puddle. The type 302 alloy plate welded with a 19 Cr-9 Ni electrode shows no crater cracking. Heliarc welding and atomic hydrogen welding of 19 Cr-9 Ni materials, stabilized with columbium, also result in crater cracking. Typical cracks with this type of welding on columbium-bearing 19 Cr-9 Ni as compared to crack-free craters on 19 Cr-9 Ni material are shown in Fig. 33.

The presence of these crater cracks does not necessarily constitute a hazard since they are readily removed by grinding, and all production welding with this type of alloy should be done with proper grinding of the craters. Similar cracking is observed in 25 Cr-20 Ni Cb and 16 Cr-13 Ni-3 Mo Cb as well as carbon steel to which columbium was added and ingot iron. With the columbium-carbon ratio 10 to 1 the extent or magnitude of the cracking is directly proportional to the columbium content, and it is believed that at carbon values of near 0 per cent this type of cracking would no longer exist even with columbium values of the order of 1 per cent. This detrimental effect of columbium on weld quality justifies its limitation to 1.00 per cent maximum in stabilized 19 Cr-9 Ni alloys.

The replacement of columbium with

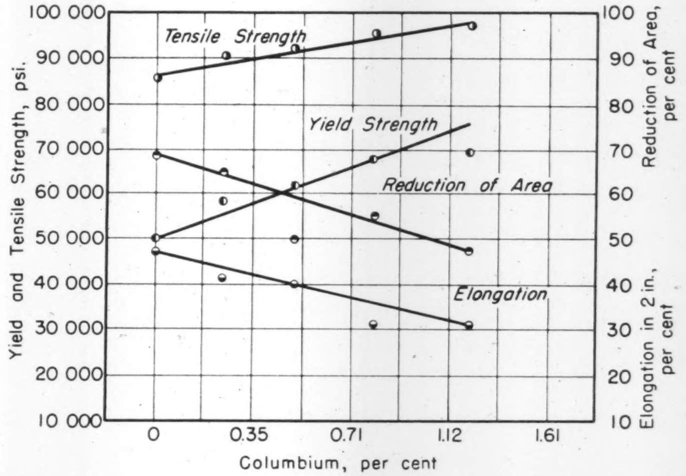
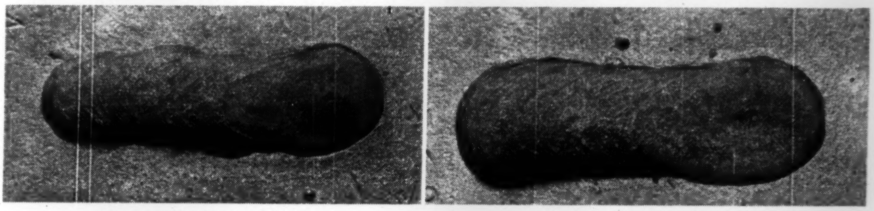
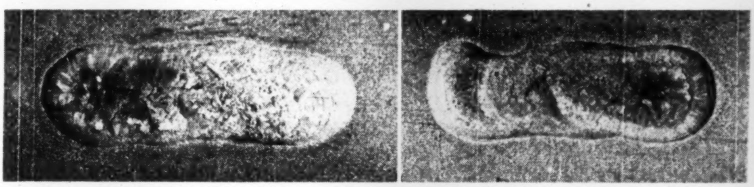


FIG. 31.—Effect of Increasing Columbium Content on Room-Temperature Physical Properties.



19 Cr-9 Ni Weld Metal 19 Cr-9 Ni Cb Weld Metal

Crater on Type 302 Base Material

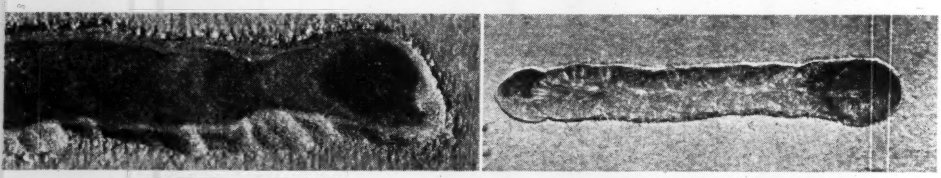


19 Cr-9 Ni Weld Metal 19 Cr-9 Ni Cb Weld Metal

Crater on Type 347 Base Material

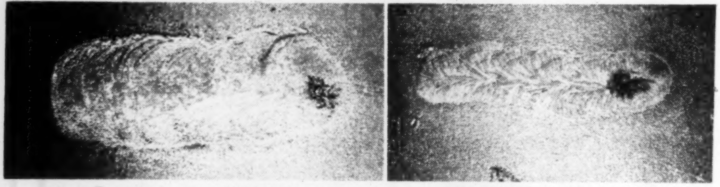
FIG. 32.—Crater Cracking Produced by Columbium Present in Either the Electrode of the Base Material. Columbium-Free Crater Shows No Cracking (actual size).

other stabilizing or carbide-forming elements so far has not been entirely successful. Investigation of the addition of tungsten, molybdenum, and cobalt resulted in excessive sigma formation on aging and, in general, less desirable microstructures than those obtained with the conventional additions. Small amounts of boron added to a 19 Cr-9 Ni Cb weld metal at low columbium values appears to show some promise, although the data at present are insufficient to permit any definite conclusions.



Atomic Hydrogen Weld Bead Heliarc Weld Bead

Type 302 Base Material



Atomic Hydrogen Weld Bead Heliarc Weld Bead

Type 347 Base Material

FIG. 33.—Craters of Atomic Hydrogen and Heliarc Beads on Type 302 and Type 347 Plate (CIX).

FERRITIC WELDING APPLIED TO DISSIMILAR WELDS

It has generally been the rule in welding that because of dilution problems a higher alloy should not be welded with a lower one. The successful welding of an austenitic material to a ferritic one with a ferritic electrode seems to break this long-standing rule. It would be expected that dilution at the higher alloy face would result in brittleness and cracking. The lime-ferritic type coating together with the use of low-carbon contents in the electrode has been found to make this type welding possible, provided high-carbon austenitic base materials are avoided.

This technique has been investigated by the Babcock & Wilcox Co. for a possible answer to two distinct problems found in joining dissimilar materials. The first of these is the carbon migration problem. By the use of a low-carbon deposit of low-chromium weld metal between the normal 0.15 per cent maximum carbon—say $2\frac{1}{4}$ Cr alloy—and an austenitic material, the carbon migration potential is reduced and the possibility of this type of failure under repeated stresses is measurably lessened. Secondly, in some applications such as austenitic support attachments to superheater tubes, it removes the point of high stress due to expansion from the colder face of the tube to a higher temperature zone having less temperature cycling and consequently lower operating stress. Also, as previously discussed, it improves the metallurgical transition between the high alloy and the low one. This results in advantages in the expansion stress problem and eliminates the sharp transition from a hard to a soft zone at the fusion line.

Tests of the use of this welding technique are of interest and will be briefly described. Original low carbon $2\frac{1}{4}$ Cr-1 Mo electrodes were made up using ingot iron core wire as a base with the necessary alloying supplied by the coating. A check of the dilution characteristics was made by means of "pad welding" on a cast 25 Cr-20 Ni plate. Layers of $\frac{1}{16}$ in. thickness were machined from the pad and chemical analysis made. At the same time each layer was checked for its hardness. Table X shows the results of this test. These data show a gradual change from an austenitic to a ferritic structure at the fusion line. Such a gradual dilution will succeed in distributing the expansion stresses over a band of reasonable width instead of concentrating them at a very narrow line as occurs when austenitic welding is employed.

TABLE X.—CHEMICAL ANALYSIS (PER CENT) OF SUCCESSIVE $\frac{1}{16}$-IN. LAYERS OF LOW-CARBON $2\frac{1}{4}$ CHROMIUM-1 MO WELD PAD DEPOSITED ON A 25 CR-20 NI CASTING.

Layer	C	Cr	Ni	Brinell Hardness Number
First	0.05	3.15	0.37	227
Second	0.04	3.36	0.56	269
Third	0.03	4.27	1.15	286
Fourth	0.05	5.08	3.01	310
Fifth	0.08	9.18	5.21	262
Sixth	0.12	14.32	9.93	201
Seventh	0.20	19.00	12.87	192

Table XI gives the chemical composition and machanical properties after various heat treatments, and the hardness of low-carbon $2\frac{1}{4}$ Cr-1 Mo weld deposits.

To check the expansion theory discussed above, additional quench tests similar to those described above were made on 25 Cr-12 Ni lugs welded to a $2\frac{1}{4}$ Cr tube with both the low-carbon chromium electrode and with 25 Cr-20 Ni electrodes. Figure 15 again shows an austenitic alloy lug welded to a $2\frac{1}{4}$ Cr tube with 25 Cr-20 Ni electrodes after 55 quenches from 1150 F. Figure 15 shows a section of this weld and illustrates the type of failure. In comparison with these results, Fig. 16 shows a low-carbon $2\frac{1}{4}$ Cr-1 Mo weld after quench testing. No failures of this weld were observed.

Metallographic examination of the previously described welds after test illustrates the difference in potential carbon migration of the two type welds. Figures 34 and 35 compare the two at 250 magnifications.

Elevated temperature stress rupture studies of the low-carbon $2\frac{1}{4}$ Cr weld metal have been made and are given in the following section on stress-rupture studies of weld metals.

TABLE XI.—CHEMICAL ANALYSIS, MECHANICAL PROPERTIES AND HARDNESS OF LOW-CARBON $2\frac{1}{4}$ CHROMIUM-1 MO WELD METAL.

Chemical Analysis, per cent					
C	Mn	Si	Cr	Ni	Mo
0.05	0.57	0.49	2.40	0.08	0.88

Condition	Yield Point, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Brinell Hardness Number
As welded	83 750	99 500	22.0	57.0	205
Stress relieved at 1050 F.	68 500	98 000	24.0	65.9	185
Stress relieved at 1350 F.	58 000	77 500	28.0	73.3	160

STRESS-RUPTURE PROPERTIES OF AUSTENITIC AND FERRITIC WELD METALS

The usefulness of weld metals and welded joints at elevated temperature and relatively high service stresses is largely a function of the behavior of the weld metal or welded joint under stress-rupture type of loadings. There is little published data available on the stress-rupture properties of austenitic or ferritic weld metals. In order to fill this need for information, a considerable amount of testing of the stress-rupture properties of weld metals has been carried on in the laboratories of the Babcock & Wilcox Co.

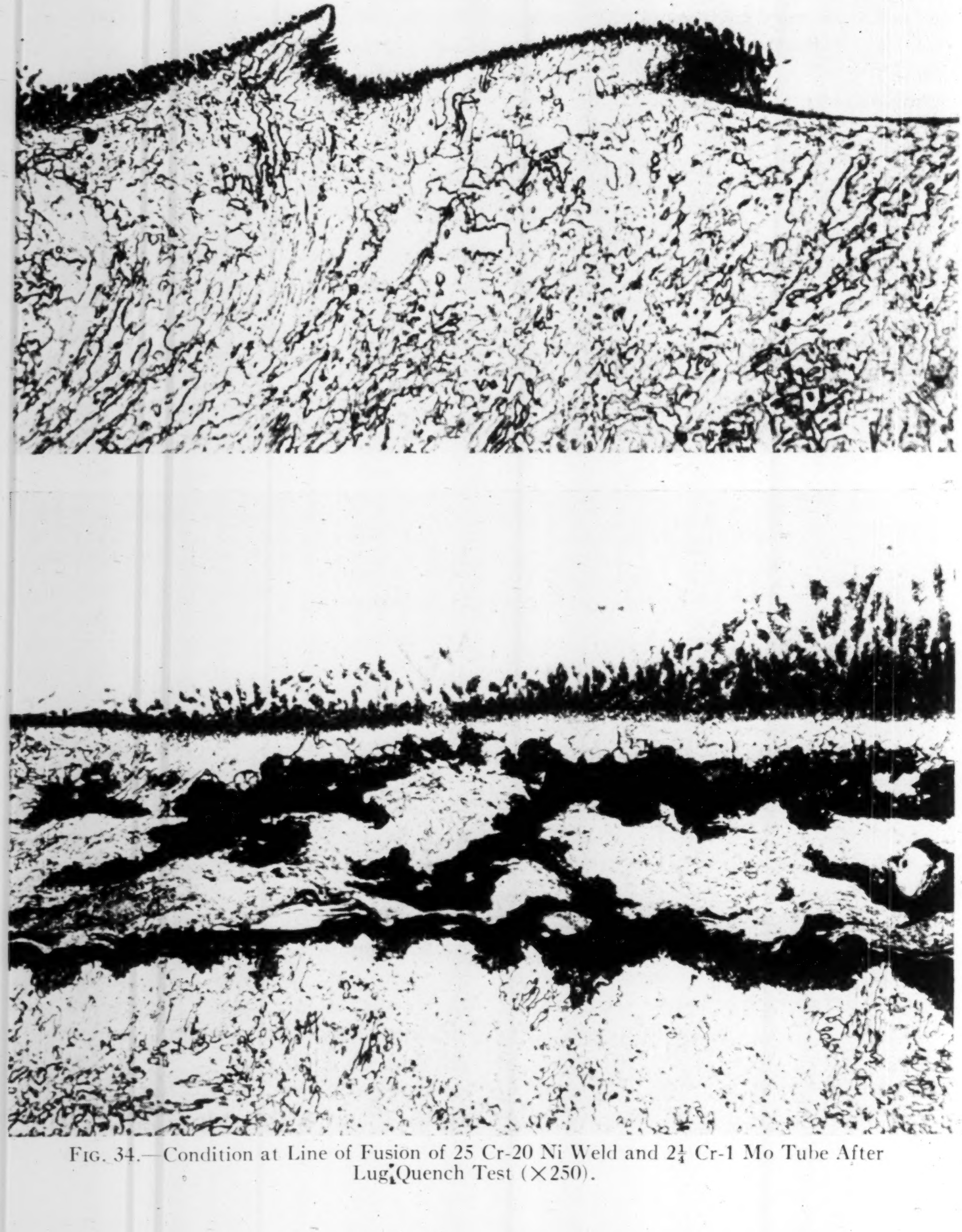
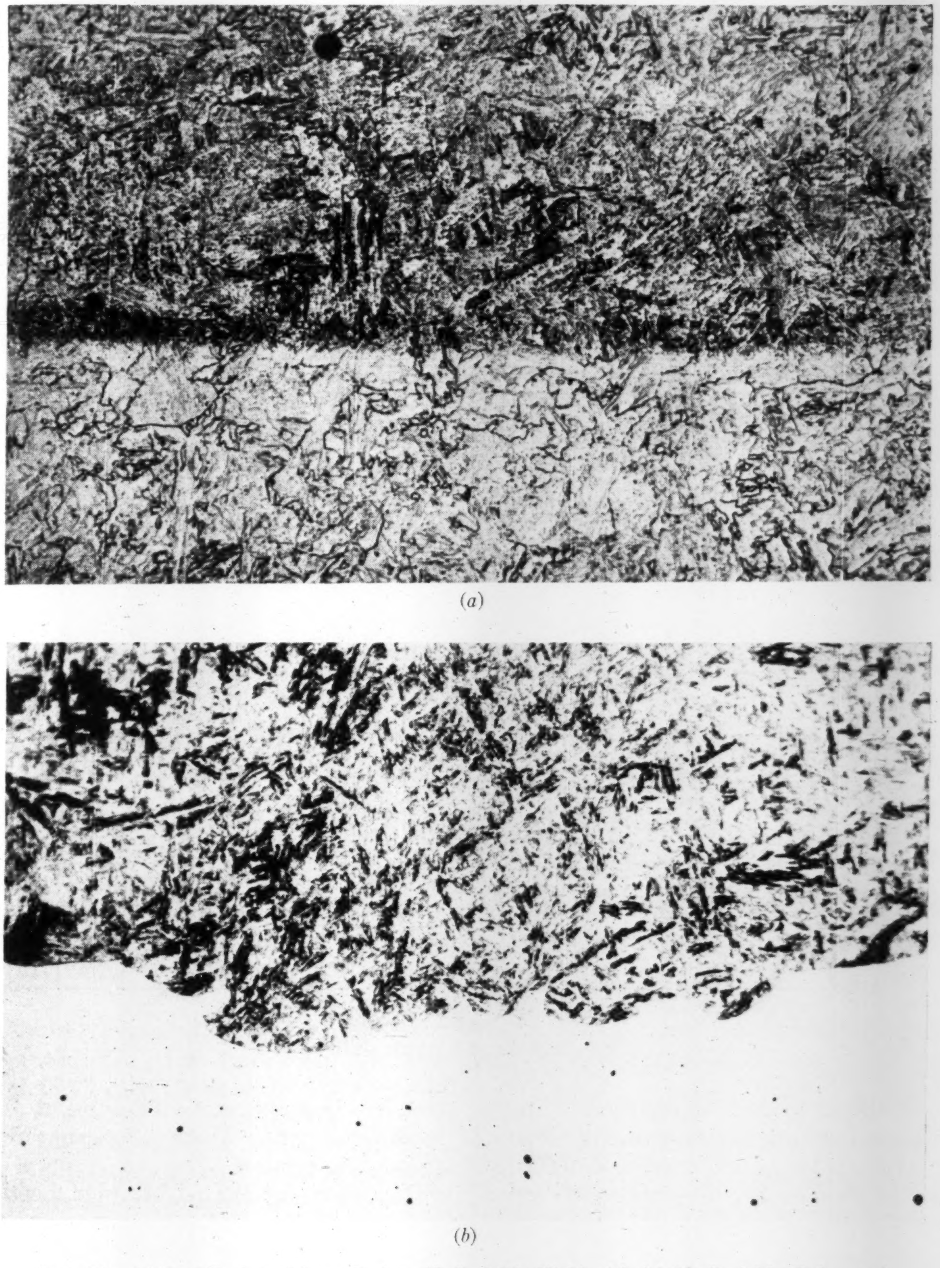


FIG. 34.—Condition at Line of Fusion of 25 Cr-20 Ni Weld and $2\frac{1}{4}$ Cr-1 Mo Tube After Lug Quench Test (×250).



(*a*)

(*b*)

FIG. 35.—Line of Fusion of Low Carbon $2\frac{1}{4}$ Cr-1 Mo Weld with (*a*) $2\frac{1}{4}$ Cr-1 Mo Tube, and (*b*) Austenitic 25 Cr-12 Ni Lug (×250)

For purposes of this discussion the log-stress—log-rupture time plot for 25 Cr-20 Ni and 19 Cr-9 Ni Cb all weld metal specimens at 1200 F. and for $2\frac{1}{4}$ Cr-1 Mo at 0.05 and 0.08 per cent carbon level at 1000 F. are shown in Fig. 36.

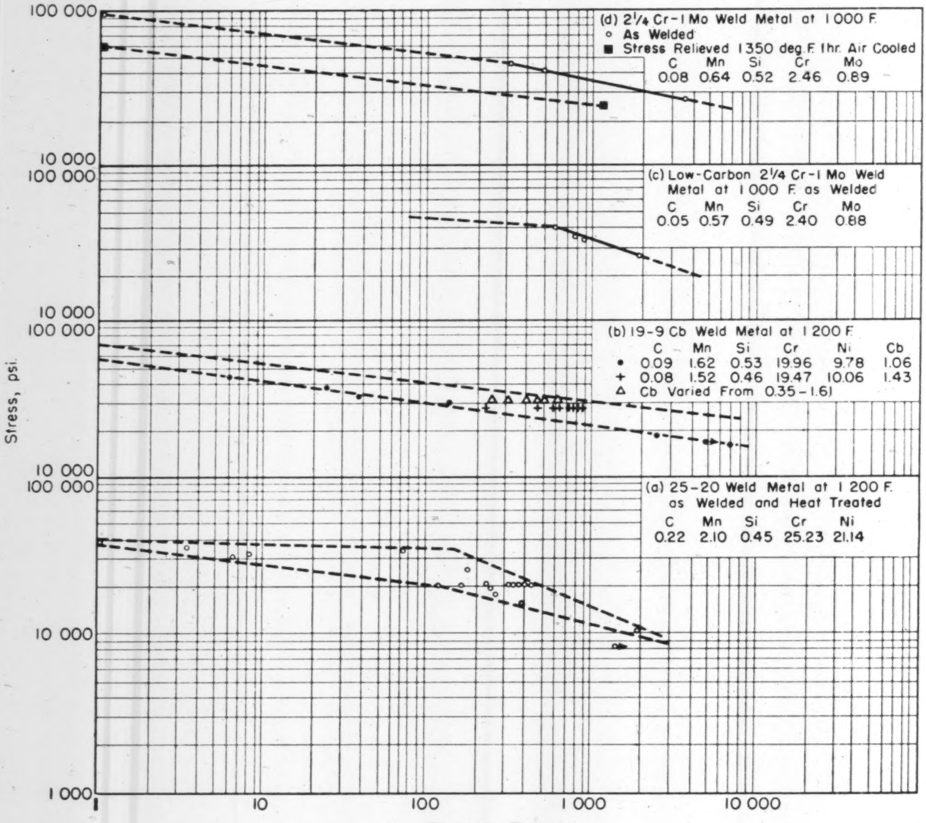


FIG. 36.—Stress Rupture Curves.

In the 25 Cr-20 Ni curve there is an apparent inflection in the spread of the point at about 160 hr. and a stress of 20,000 psi. The 19 Cr-9 Ni Cb weld metal stress-rupture curve does not indicate an equal inflection but appears to retain a constant slope at a higher stress. This difference is believed to be the result of a different mode of formation of sigma constituent. Because of this exhibited higher strength, the tendency of this alloy weld metal to form less sigma, and because it is less susceptible to micro-fissuring the 19 Cr-9 Ni Cb electrode was selected as the most suitable for the welding of dissimilar metals where austenitic filler metal is used and where high - temperature strength is required.

The charts for $2\frac{1}{4}$ Cr-1 Mo weld metal having 0.05 and 0.08 per cent carbon, respectively, at 1000 F. are shown in Fig. 36. At 40,000 psi. stress the two alloys have about the same life while the extrapolated stress for rupture in 10,000 hr. is 20,000 psi. in the case of the 0.08 per cent carbon $2\frac{1}{4}$ Cr and about 16,000 psi. for the 0.05 per cent carbon material.

In addition to these all weld metal tests some were made on dissimilar metal specimens transverse to the weld. Several fractured specimens are shown in Fig. 37. These specimens are tests of 18 Cr-8 Ni Cb welded to $2\frac{1}{4}$ Cr-1 Mo with 19 Cr-9 Ni Cb weld metal.

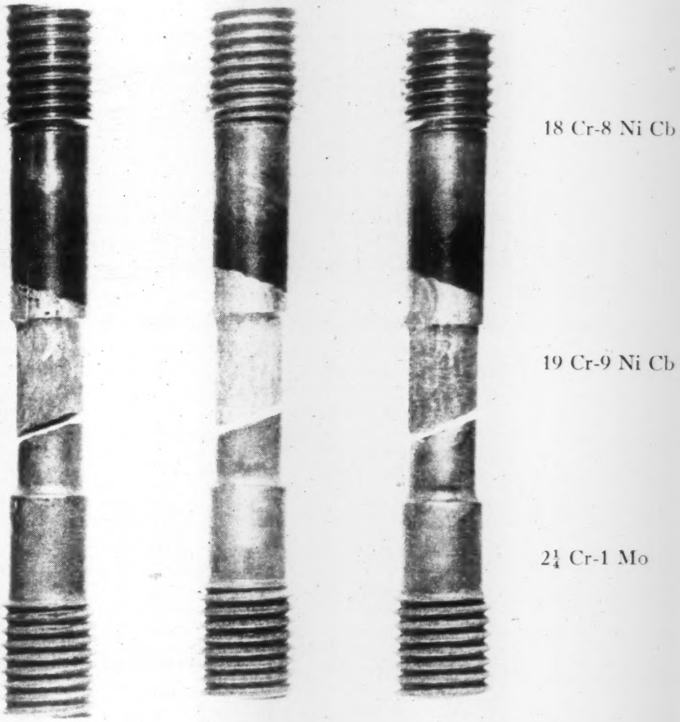


FIG. 37.—Three Fractured Dissimilar Metal Stress Rupture Specimens Run at 1150 F.

The test temperature was 1150 F. The fracture occurred in the same manner as in fatigue and thermal quenching tests. The data obtained on these specimens are plotted in Fig. 38. For comparison, a curve for wrought $2\frac{1}{4}$ Cr steel is included which was interpolated from Babcock & Wilcox Tube Co. *Bulletin 6-E.* As the stress decreased and fracture time increased, there is a marked difference in the slope of the two curves. This is believed to be the result of stress set up by differential expansion plus the combined effects of carbon migration and accelerated oxidation, or, in other words, these failures follow the same pattern as do fatigue tests.

DISCUSSION

Inherent Stresses:

From the above-described work together with reviews of the literature and in consideration of actual service experience it seems reasonable to enumerate the factors which will govern the successful operation of a dissimilar metal joint in service.

The first and the basic one is the inherent expansion differential between an austenitic and a ferritic material. For a quick comparison, an 18 Cr-8 Ni alloy has a coefficient of linear expansion in the temperature range of 70 F. to 1300 F. of 10.75×10^{-6} in. per in. deg. Fahr A ferritic $2\frac{1}{4}$ Cr-1 Mo alloy has, for the above temperature range, a coefficient of 7.88×10^{-6}. Obviously, upon heating a dissimilar metal joint which has been welded with an austenitic weld metal, for instance 19 Cr-9 Ni Cb, the expansion stresses, produced as a result of the 36 per cent difference, will reach yield point dimensions at a narrow band along the junction of the two materials unless the lower strength one yields or creeps. This has been shown by measurable increases in diameter of

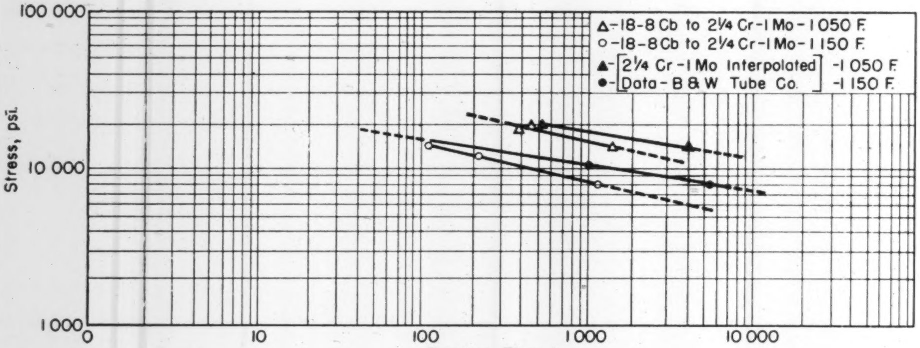
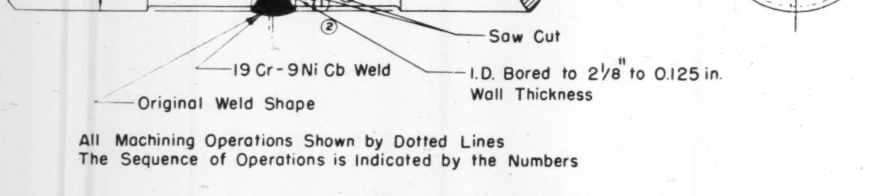


FIG. 38.—Stress Rupture Curves for Transverse Dissimilar Metal Weld Specimen at 1050 and 1150 F

TABLE XII.—RESIDUAL STRESS IN DISSIMILAR WELDS.

Gage Location	Observed Stress, psi.			Observed Stress Corrected for Poisson's Ratio		
	As Welded	Water Quench from 1050 F.	104 Cycles of Quenching	As Welded	Water Quench from 1050 F.	104 Cycles of Quenching
1	330	2460	2607	2418	9909	12061
2	6960	33030	31515	7059	33768	32297
3	....	22350				
4		25950				


0.560"
2
2
Weld and Tube-Machine to 2.400 in. O.D.
SR-4 Gages Mounted as Shown
2.500"
Saw Cut
18 Cr-8 Ni Cb
5
6
4
8
7
Gage No. 1
Gage No. 2
Gage No. 3
Gage No. 4
2¼ Cr-1 Mo
1
2
3
Saw Cut
19 Cr-9 Ni Cb Weld
Original Weld Shape
I.D. Bored to 2⅛" to 0.125 in.
Wall Thickness
Gage No. 1
Gage No. 2
Gage No. 3
Gage No. 4

All Machining Operations Shown by Dotted Lines
The Sequence of Operations is Indicated by the Numbers

tubular specimens of dissimilar joints where the ferritic side has been operating in the low creep temperature range. Figure 8 shows very plainly the strain lines in a tubular fatigue specimen which are believed to be present largely because of the expansion stresses.

A study of the magnitude of these stresses has been made by means of SR-4 type gages and the removal of relaxation specimens from tubular specimens across the junction line. Several specimens have been checked, one as-welded, a second after a single quench from 1050 F., and a third after 104 cycles of heating and quenching in the cyclic-quench apparatus. The results of these observations are given in Table XII. The as-welded sample shows a reasonable stress of approximately 6900 psi. The single quench specimen showed stresses at a location just off the fusion line, in the circumferential direction, and in the ferritic alloy of 33,000 psi. The specimen which has had 104 quenches showed, at the same location, an approximately equal stress of 31,500 psi. Within the limits of error of this method of analysis, it is evident that these expansion stresses together with working stresses place the operating stresses on the ferritic side of transition welds well over the yield point. The type of failure shown in the tests described in this report should, therefore, be expected and especially where temperature cycling occurs, where movement of the ferritic alloy cannot take place, and where the metallurgical phenomena described in this paper are present.

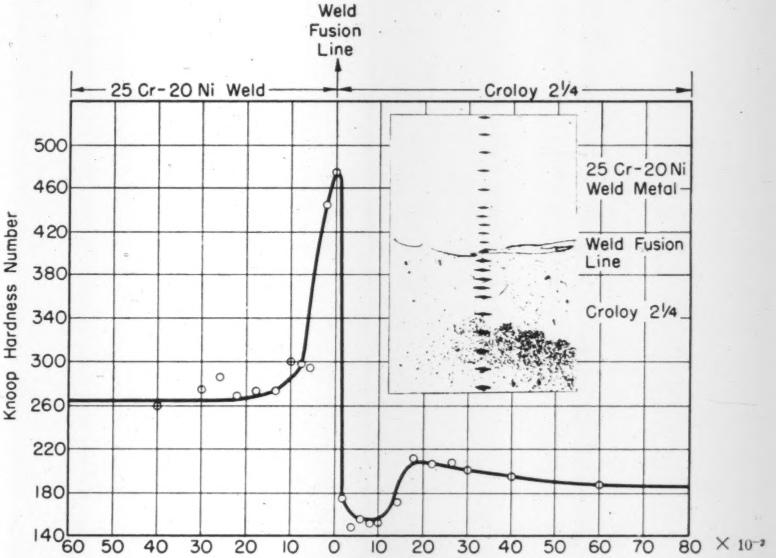
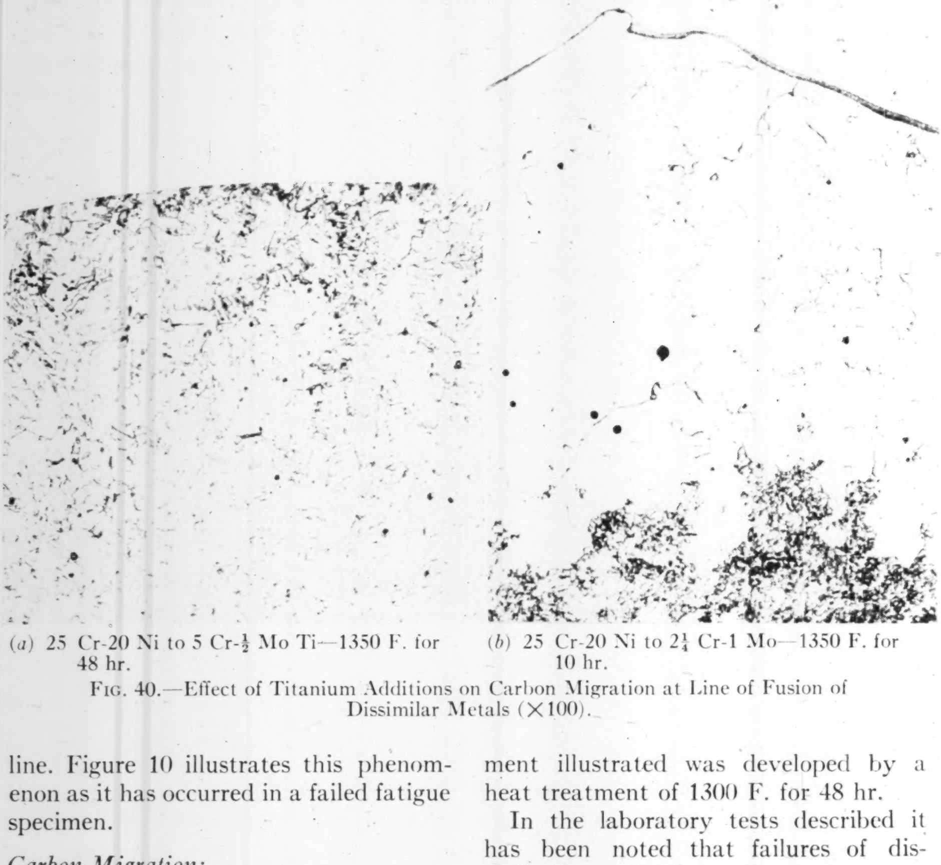


FIG. 39.—Microhardness Traverse of Dissimilar Metal Weld Specimen Heated 48 hr. at 1300 F.

Oxidation:

Oxidation limits of the low-chromium ferritic materials, particularly $2\frac{1}{4}$ Cr-1 Mo which because of its high creep strength is of greatest interest, is generally assumed to be 1150 to 1175 F., when operating under low stress. This work points out that when ferritic to austenitic joints are operating at or near the oxidation limit, the stress concentrations along the fusion line destroy or cracks the protective scale of the ferritic material resulting in oxide penetration or notching at or near the fusion line. Figure 10 illustrates this phenomenon as it has occurred in a failed fatigue specimen.



(*a*) 25 Cr-20 Ni to 5 Cr-½ Mo Ti—1350 F. for 48 hr.

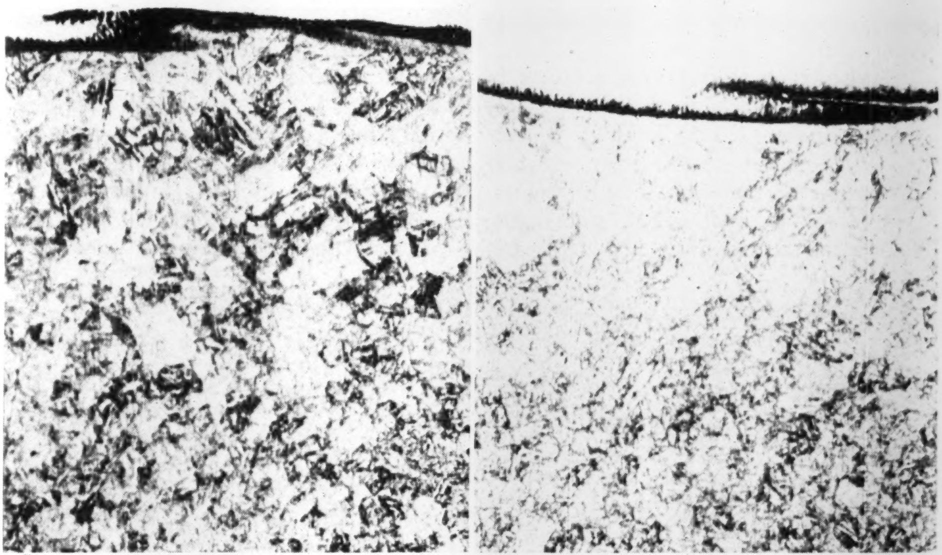
(*b*) 25 Cr-20 Ni to 2¼ Cr-1 Mo—1350 F. for 10 hr.

FIG. 40.—Effect of Titanium Additions on Carbon Migration at Line of Fusion of Dissimilar Metals (×100).

Carbon Migration:

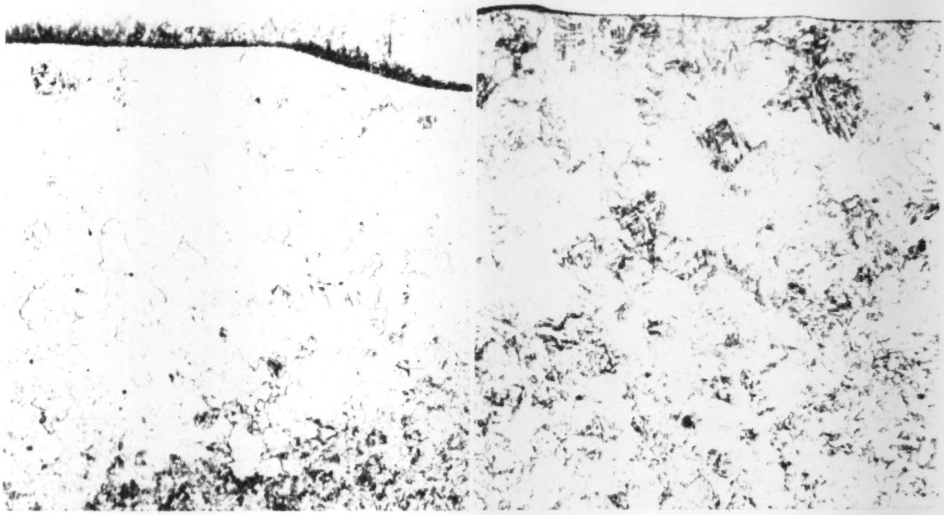
When an austenitic material is joined to a ferritic one and heated in the temperature range considered throughout this paper, carbon movement usually takes place producing a hard, thin, high-carbon layer in the austenitic material adjacent to the ferritic line of fusion. In the ferritic material a decarburized area adjacent to the high carbon line usually exists. Figure 39 shows a micro-hardness survey of an austenitic material welded to a ferritic material. Notice the sharp increase in hardness at the fusion line and the comparative softness of the adjacent decarburized layer. The carbon movement illustrated was developed by a heat treatment of 1300 F. for 48 hr.

In the laboratory tests described it has been noted that failures of dissimilar metal joints frequently occur adjacent to the hard zone and in the low-carbon ferrite, probably as a result of its comparatively low strength. In many service failures, the presence of the decarburized layer has been lacking although there has generally existed in the samples examined a carbon concentration line at the fusion line. It may be that continuous heating over a long period of time will result in re-



(*a*) 19 Cr-9 Ni Cb to $2\frac{1}{4}$ Cr-1 Mo—1050 F. for 1000 hr.

(*b*) 25 Cr-20 Ni to $2\frac{1}{4}$ Cr-1 Mo—1050 F. for 1000 hr.



(*c*) 19 Cr-9 Ni Cb to $2\frac{1}{4}$ Cr-1 Mo—1150 F. for 1000 hr.

(*d*) 25 Cr-20 Ni to $2\frac{1}{4}$ Cr-1 Mo—1150 F. for 1000 hr.

FIG. 41.—Effects of Heating on Carbon Migration at Line of Fusion of Dissimilar Metals (×100).

carburization of the low-chromium material once a state of equilibrium is reached at the fusion line. This condition may be more readily established where the assembly is operating at comparatively low stresses.

Since carbon migration is a factor in the serviceability of dissimilar metal welds and since it may possibly be controlled by suitable carbide stabilization of the ferritic material, some investigation of this possibility has been made. This series of tests has included additions of titanium, vanadium, columbium, nickel, manganese, silicon, and molybdenum to the ferritic side of the weld. The results of these tests are given below:

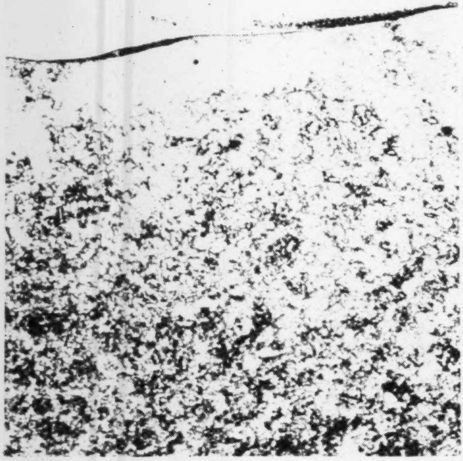


FIG. 42.—Line of Fusion of 25 Cr-20 Ni to 2¼Cr-1 Mo Heated at 1350 F. for 1 hr. (×100)

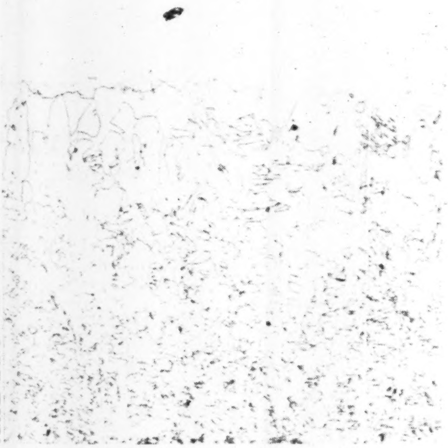


FIG. 43.—Effect of Vanadium Additions on Carbon Migration at Line of Fusion of Dissimilar Metals (×100). 25Cr-20 Ni to 2¼ Cr V—1350 F. for 48 hr.

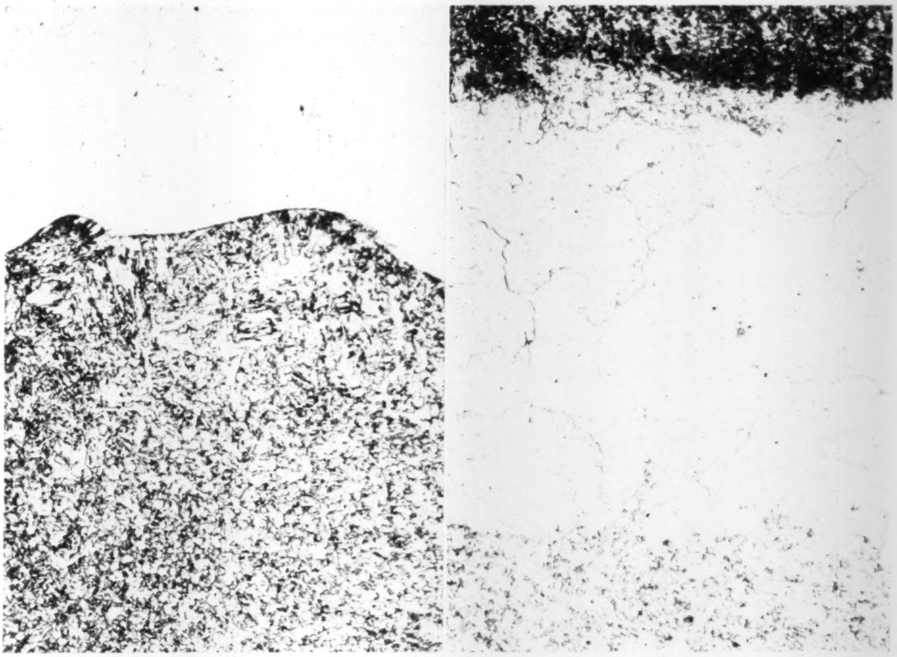
Titanium Additions to the Ferrite:

The effect of titanium on carbon migration was investigated on some wrought 5 Cr-0.5 Mo Ti material of the following analysis:

	Ladle Analysis, per cent	Check Analysis, per cent
Carbon	0.09	0.09
Manganese	0.48	0.49
Sulfur	0.016	0.016
Phosphorous	0.016	0.016
Silicon	0.33	0.31
Chromium	4.95	4.82
Molybdenum	0.52	0.50
Titanium	0.46	0.47

Both 19 Cr-9 Ni Cb and 25 Cr-20 Ni weld metal were deposited on sections of this material and subjected to 1000 hr. at 1050 F. and 1150 F., and another specimen was subjected to a 48-hr. heat treatment at 1350 F. There was no carbon migration noted in any of the specimens tested. Figure 40 (*a*) shows the line of fusion of the specimen welded with 25 Cr-20 Ni weld metal and heated for 48 hr. at 1350 F. A shorter exposure at the same temperature of a dissimilar joint involving unstabilized 2¼ Cr-1 Mo is shown in Fig. 40 (*b*). The mechanism of carbon migration as affected by temperature and time is illustrated in Figs. 41 to 46, inclusive. Figures 41 (*a*) and 41 (*b*) show the effects of heating dissimilar joints between 2¼ Cr-1 Mo unstabilized and 19 Cr-9 Ni Cb and 25 Cr-20 Ni weld metals, respectively. Definite evidence of carbon depletion of the 2¼ Cr material may be noted adjacent to the 19 Cr-9 Ni Cb weld metal with some carburization of the austenite. The 25 Cr-20 Ni weld showed considerably

less carbon depletion and practically no diffusion of carbon into the austenite. At 1150 F. the amount of decarburization of the $2\frac{1}{4}$ Cr-1 Mo is substantially greater in both cases as shown in Figs. 41 (*c*) and 41 (*d*), respectively. Short-time exposures of a 25 Cr-20 Ni weld metal to $2\frac{1}{4}$ Cr-1 Mo unstabilized joint for 1 and 10 hr. at 1350 F. are shown in Figs. 42 and 40 (*b*), respectively. The degree of decarburization and carbon diffusion into the austenite appears to be a direct function of time at temperature. The rate at which such decarburization occurs appears to be primarily a function of temperature.



(*a*) 25 Cr-20 Ni to 5 Cr-$\frac{1}{2}$ Mo Cb—1350 F. for 48 hr.

(*b*) 5 Cr-$\frac{1}{2}$ Mo Cb to $2\frac{1}{4}$ Cr-1 Mo—1350 F. for 48 hr.

FIG. 44.—Effect of Columbium Additions on Carbon Migration at Line of Fusion of Dissimilar Metals (×100).

Effect of Vanadium Addition to the Ferrite:

Vanadium stabilized wrought material was obtained from a heat of steel having the following chemical analysis:

	Actual, per cent
Carbon	0.07
Manganese	0.19
Silicon	0.13
Chromium	2.46
Molybdenum	1.13
Vanadium	0.43

A 25 Cr-20 Ni weld metal layer was deposited on this material and subjected to a 48-hr. heating at 1350 F. No carbon migration was noted in this test, indicating the effectiveness of vanadium as a carbide stabilizer. Figure 43 illustrates these results.

Effect of Columbium Additions to the Ferrite:

Since no columbium-bearing wrought material was available, an experimental 5 Cr electrode was produced with columbium additions being supplied by the coating. The molybdenum content was raised simultaneously to the $2\frac{1}{4}$ Cr level. Analysis of the undiluted weld was metal as follows:

Carbon, per cent	0.05
Manganese, per cent	0.96
Silicon, per cent	0.60
Chromium, per cent	5.39
Molybdenum, per cent	1.22
Columbium, per cent	0.67

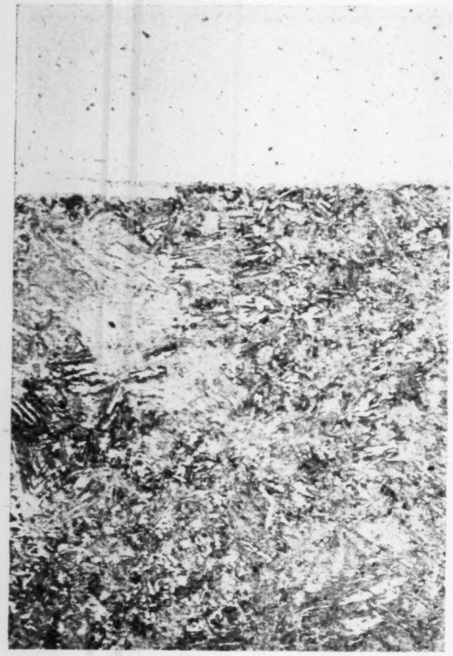


FIG. 45.—Effect of Nickel Additions on Carbon Migration at Line of Fusion of Dissimilar Metals (×100). 25 Cr-20 Ni to $2\frac{1}{4}$ Cr-1 Mo Ni—1350 F. for 48 hr.

A pad of weld metal was deposited on $2\frac{1}{4}$ Cr plate material and this weld metal was covered with several layers of 25 Cr-20 Ni weld metal. The specimen was then subjected to a 48-hr. heating at 1350 F. Examination of the weld junction between the 25 Cr-20 Ni and 5 Cr Mo Cb indicated no carbon migration or diffusion into the austenite as shown in Fig. 44 (*a*). It was noted, however, that considerable diffusion of carbon occurred from the unstabilized $2\frac{1}{4}$ Cr wrought material to the stabilized 5 Cr weld metal as shown in Fig. 44 (*b*).

Effects of Manganese and Nickel Additions to the Ferrite:

In order to study the effect of manganese and nickel additions to $2\frac{1}{4}$ Cr material, special low-carbon $2\frac{1}{4}$ Cr electrodes were prepared with nickel and manganese additions made through the coating. Analyses of the undiluted weld metals are listed below:

	Nickel	Manganese
Carbon, per cent	0.04	0.03
Manganese, per cent	0.40	3.00
Silicon, per cent	0.31	0.29
Chromium, per cent	2.91	2.86
Molybdenum per cent	1.13	1.07
Nickel, per cent	2.53	....

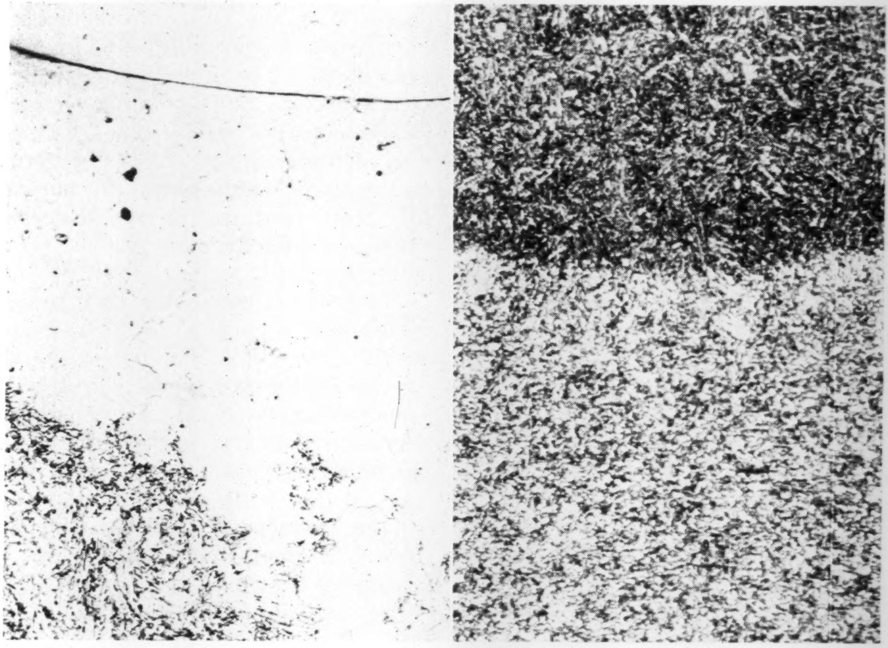
Weld pads were prepared on $2\frac{1}{4}$ Cr base material in a manner similar to that used for the columbium-stabilized electrode. A 48-hr. heating at 1350 F. produced no noticeable carbon migration at the ferrite-austenite interface. Figure 45 shows the condition observed with the nickel-bearing weld metal. The addition of manganese showed a similar condition at the line of fusion.

Effects of Silicon Addition to the Ferrite:

A high-silicon low-carbon $2\frac{1}{4}$ Cr analysis was prepared by using an electrode depositing a low-carbon, high-silicon weld metal. Analysis of the deposit was as follows:

Carbon, per cent	0.04
Manganese, per cent	1.89
Silicon, per cent	1.50
Chromium, per cent	3.03
Molybdenum, per cent	1.11

Test specimens were prepared as above and subjected to a 48-hr. heating at 1350 F. Figure 46 (*a*) shows that considerable migration of carbon from the high-silicon ferrite to the austenite occurred, in spite of the unusually high manganese content of the weld metal. No carbon diffusion from the low-silicon $2\frac{1}{4}$ Cr base plate to the high-silicon weld was noted as shown in Fig. 46 (*b*).



(*a*) 25 Cr-20 Ni to $2\frac{1}{4}$ Cr-1 Mo Si—1350 F. for 24 hr.

(*b*) $2\frac{1}{4}$ Cr-1 Mo Si to $2\frac{1}{4}$ Cr-1 Mo—1350 F. for 48 hr.

FIG. 46 —Effect of Silicon Additions on Carbon Migration at Line of Fusion of Dissimilar Metals (×100).

The Effect of Additional Molybdenum in the $2\frac{1}{4}$ Cr-1 Mo Composition:

A low-carbon $2\frac{1}{4}$ Cr electrode was prepared with an addition of molybdenum to the final analysis through the electrode coating. Analysis of the deposit was as follows:

Carbon, per cent	0.04
Manganese, per cent	1.30
Silicon, per cent	0.31
Chromium, per cent	2.94
Molybdenum, per cent	1.90

Test specimens were prepared and heat treated as in the previous investigations. Figure 47 shows that the additional molybdenum did not prevent carbon migration at 1350 F.

Based on the study of the effect of addition of alloying elements to a ferritic material, it appears that the rate or degree of carbon migration from the ferrite to the austenite may substantially be reduced by additions of columbium, titanium, vanadium, nickel, or manganese listed in the order of their effectiveness. Silicon additions did not appear to be effective although the silicon level was relatively low and there is always a degree of segregation in a weld deposit, particularly when an alloying element is added through the electrode coating. The effect of the microstructure of the ferritic member of the dissimilar joint has not been investigated, although it is believed that the effect of prior heat treatment of the ferritic material would be destroyed in the process of welding, at least in that area immediately adjacent to the line of fusion.

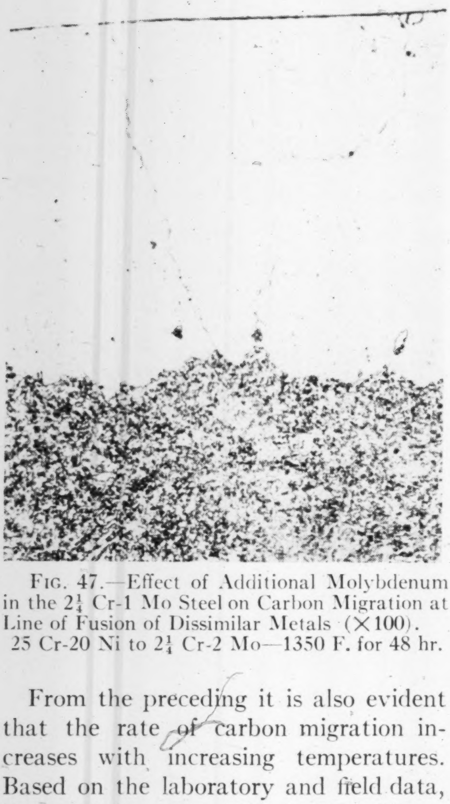


FIG. 47.—Effect of Additional Molybdenum in the 2¼ Cr-1 Mo Steel on Carbon Migration at Line of Fusion of Dissimilar Metals (×100).
25 Cr-20 Ni to 2¼ Cr-2 Mo—1350 F. for 48 hr.

From the preceding it is also evident that the rate ~~of~~ carbon migration increases with increasing temperatures. Based on the laboratory and field data, it would be very difficult to select a limiting temperature to which a dissimilar joint may be subjected for an indefinitely long time without showing a marked carbon depletion of unstabilized ferrite adjacent to the weld junction.

HEAT TREATMENT

Any heat treatment given a weld joining an austenitic to a ferritic material cannot improve the serviceability of the assembly. In unstabilized ferritic material, carbon migration will be produced. With any material combination, high stresses are established which have been shown to be of yield point magnitude at 1050 F., and higher temperatures should increase these stresses. The expansion differential of the two materials makes relief of stress difficult or impossible. Heat treatment in the tempering range of the ferritic alloy produces sigma in the austenitic weld.

The stress-relieving treatment usually considered necessary for hardenable, ferritic, low-chromium alloys is not necessary where welding is done with low-hydrogen coatings and where the service temperature is over 950 F. For example, the heat-affected zone of a 2¼ Cr-1 Mo plate welded with 19 Cr-9 Ni is reduced in hardness from 320 Brinell hardness number as-welded to under 220 Brinell hardness number after 40 hr. at 1050 F. (See Appendix.)

CONCLUSIONS

It is difficult to estimate the degree of success to be expected of a dissimilar metal joint in service. The work reported here, together with service experience, points to several factors which, when controlled, will measurably reduce the possibility of failure. These will be briefly enumerated:

1. The weld metal used may be either austenitic or ferritic, but each should demand an exacting control of the electrode itself and of the technique employed. This control should include careful shop tests of the electrodes to insure absence of fissuring. Careful procedures and welding details must be extablished. Welding techniques in

production should be carefully controlled to assure removal of crater cracking or other mechanical defects.

The cover beads should be removed flush to the surface and all other stress raisers such as notches and undercuts carefully removed when they are near the fusion line.

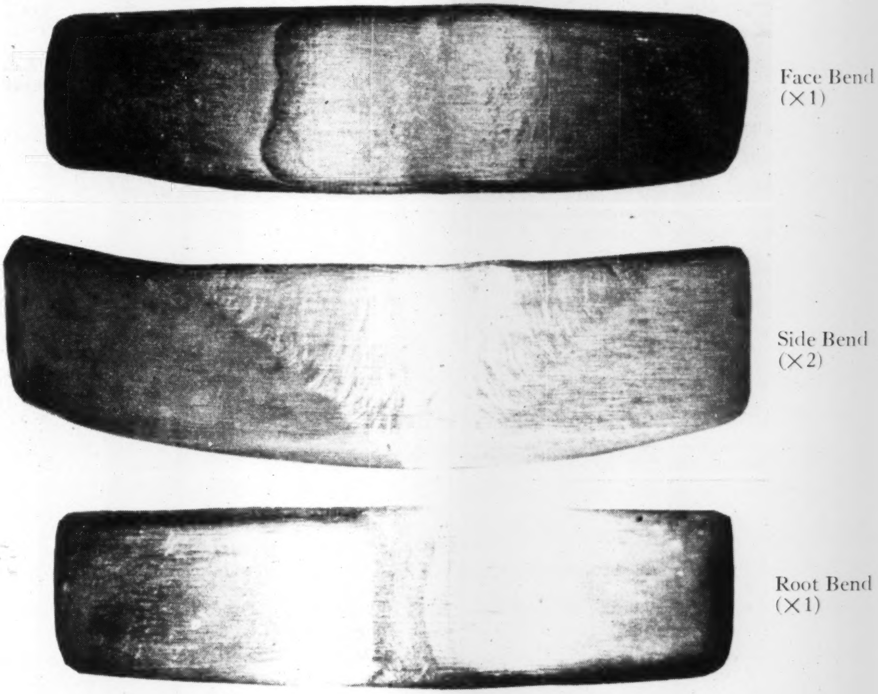
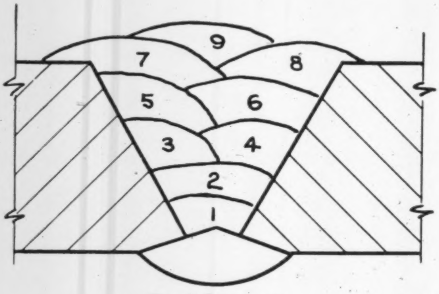


FIG. 48.—Procedure Qualification Bends on 18 Cr-8 Ni Cb to $2\frac{1}{4}$ Cr-1 Mo Welded with 19 Cr-9 Ni Cb Electrode in the As-Welded Condition.

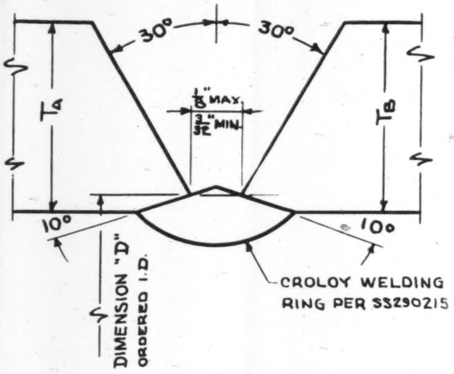
2. No post-welding heat treatment need be given welds joining ferritic to austenitic materials when the ferritic material does not show as-welded hardnesses over 350 Brinell hardness number, and when service temperatures exceed 950 F. This will reduce carbon depletion on the ferritic side and will lessen the rate of sigma formation in the austenitic weld metal.

3. The choice of welding composition seems to be a 19 Cr-9 Ni Cb electrode for most applications. The work given above establishes reasons for this, being basically soundness and better high temperature strength. A low-chromium, low-carbon electrode shows excellent possibilities, certainly for non-pressure parts or for pressure parts when more information on its high temperature properties becomes available.

4. The fundamental cause of failure of a transition weld in a ferritic to an austenitic material is expansion and operating stresses. Other secondary causes such as oxidation fatigue, carbon migration and poor welding techniques speed these failures. The safe operating limits to be expected of a dissimilar metal weld appear to be a function



Bead Sequence

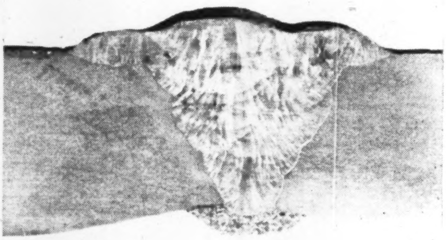

30°
30°
1/8" MAX.
3/32" MIN.
Ta
TB
10°
10°
DIMENSION "D"
ORDERED I.D.
CROLOY WELDING RING PER SS290215

Joint Detail

PASS NO.	BEAD TYPE	ELECTRODE	CURRENT
1	String	$\frac{3}{32}$ in. Diam. AISI Type 347 Lime-coated for all passes	55–56 Amps. for all passes
2	Slight Weave		
3.4	String		
5.6	Slight Weave		
7.8.9	Slight Weave		

APPLICATION

MATERIAL	OUTSIDE DIAMETER in.	WALL, in.	JOB NO.
AISI Type 347	2½	0.566	
AISI Type 347	2½	0.520	RB 50
Croloy 3M	2½	0.563	SH 7933
Croloy 3M	2½	0.566	



Section through Joint

NOTE.—Actual test was made between type 347, 2½ in. O.D. by 0.520 in. wall and Croloy 3M 2½ in. O.D. by 0.563 in. wall. (T_a-0.563 in., T_b-0.520 in.)

FIG. 49.—Metal Arc Welding of Type 347 Tubes to Croloy 3 M Tubes for Superheater Service. (A.S.M.E. Case 1022) (2½ in. O.D., Wall Thicknesses Over $\frac{3}{8}$ in. and Including $\frac{3}{4}$ in.)

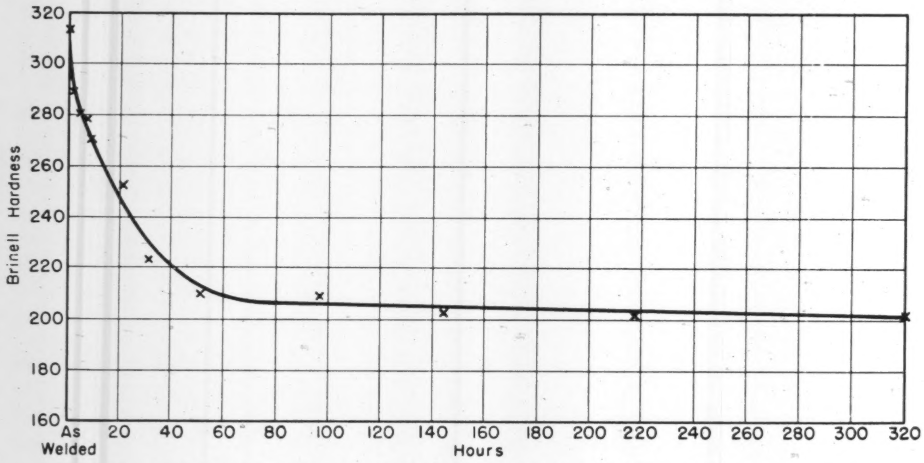


FIG. 50.—18-8 Weld Metal on 2¼ Cr-1 Mo Plate. Hardness of Heat Affected Zone After Exposure at 1050 F.

of the operating temperature and the degree of temperature cycling. This work points out that endurance limits of $2\frac{1}{4}$ Cr-1 Mo tubing welded with 19 Cr-9 Ni Cb to 18 Cr-8 Ni Cb tubing operating at metal temperatures of 1150 F. are well beyond those to be expected in normal superheater service. This presupposes that the various welding and material precautions which have been suggested have been followed.

5. Where temperature and stress conditions are such as to indicate possible failure during the normal life of the assembly, it is recommended that all cyclic temperatures be prevented at dissimilar joints. This may best be accomplished by separate heating as, for example, by Calrods or by other similar methods, applied to the outside of the joint. These may automatically be controlled so as to prevent the cooling of the weld under the approximate operating temperature range.

This procedure will refer particularly to piping. On small tubing such as in superheaters this work would indicate that this precaution is not necessary under normal operating conditions.

APPENDIX

Qualifications of welding procedure for a dissimilar metal weld were made in accordance with ASME Case 1022. The specimens were prepared from tubes of $2\frac{1}{2}$ in. outside diameter and 0.5 in. minimum wall thickness of 18 Cr-8 Ni Cb and $2\frac{1}{4}$ Cr-1 Mo welded with 19 Cr-9 Ni Cb electrodes. The process was first qualified for 25 Cr-20 Ni weld metal and was later requalified for 19 Cr-9 Ni Cb weld metal. The face, side, and root bends shown in Fig. 48 were made on an "as welded" specimen as part of the qualification procedure. Figure 49 shows a tabular sheet giving details of procedure, including weld design, bead sequence, etc. In addition to this information it was standard procedure to preheat the $2\frac{1}{4}$ Cr-1 Mo tubing to 400 F. prior to welding. The welds were not stress-relieved after welding.

In order to establish the effect of service temperature on the hardness of the heat-affected zone of a dissimilar metal weld, a series of specimens was prepared by depositing a single bead of 19 Cr-9 Ni weld metal on $2\frac{1}{4}$ Cr-1 Mo plate. The specimens were placed in a furnace at 1050 F. and heated for periods up to 320 hr. A hardness curve for these specimens is shown in Fig. 50. Notice that after only 25 hr. the major change in hardness has occurred.

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(1) O. R. Carpenter and N. C. Jessen, "Some Factors Controlling the Ductility of 25% Cr-20% Ni Weld Deposits," *Welding Journal*, Vol. 26, December, 1947, pp. 727S–740S.

(2) M. E. Holmberg, "Welding of Steels for High Temperature Service," *Welding Journal*, Vol. 28, February, 1949, pp. 141–148.

(3) A. L. Schaeffler, "Selection of Austenitic Electrodes for Welding Dissimilar Metals," *Welding Journal*, Vol. 26, October, 1947, pp. 601S–620S.

(4) W. C. Stewart, and W. G. Schreitz, "Thermal Shock and Other Comparison Tests of Austenitic and Ferritic Steels for Main Steam Piping," Presented at Spring Meeting, Am. Soc. Mechanical Engrs. (1950).

(5) R. D. Thomas, Jr., and H. C. Campbell, "The Effect of Alloying Elements on the Tensile Properties of 25 Cr-20 Ni Weld Metal," *Welding Journal*, Vol. 25, November 1946, pp. 760S–768S.

(6) H. Weisberg, "Cyclic Heating Tests of Main Steam Piping Joints Between Ferritic and Austenitic Steels—Sewaren Generating Station," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 71, August, 1949, pp. 643–664. Discussion included.

JOINT DISCUSSION OF PAPERS BY R. U. BLASER, ET AL., AND O. R. CARPENTER, ET AL.

MR. H. WEISBERG[1] (*presented in written form*).—It would appear desirable at this time to review the differences between the Babcock & Wilcox test described in the Blaser, Eberle, and Tucker paper and two similar tests recently reported, one by Public Service Electric and Gas Company,[2] and the other by the U. S. Naval Engineering Experiment Station,[3] and consider why the failure occurred here, whereas no failure was reported for either of the other tests. A re-inspection of the coupons from the Public Service test specimens shows no evidence of the intergranular oxidation phenomenon reported by Babcock & Wilcox, and although the Navy test assembly has not been sectioned, a thorough nondestructive inspection shows no failures of this type. The quench runs of both the Public Service and the Navy tests resulted in even more rapid cooling than in the Babcock & Wilcox test. Both the Public Service and the Navy test specimens were subjected to more heating and cooling cycles than the Babcock & Wilcox piece. The Public Service pipe was tested without internal steam pressure but the Navy pipe was tested at full steam pressure.

There are, however, at least three other differences as follows which make the Babcock & Wilcox test more severe and may account for the failure:

1. The Public Service and the Navy welds were post-heated to relieve welding stresses whereas the Babcock & Wilcox welds were not. Carpenter's tests indicate that post-heat treatment is detrimental in that it increases carbon migration which was the primary cause of the failure. His tests, however, were on relatively small diameter tubing. When dealing with heavy wall pipe, the benefits of stress relieving may offset the disadvantage of greater depletion of carbon in the heat-affected zone.

2. In the Babcock & Wilcox test specimen, the $1\frac{3}{4}$ in. thick ferritic pipe was reduced to $1\frac{1}{10}$ in. at the weld resulting in a physical notch and concentration of stress at a point where there already was a metallurgical notch due to carbon depletion. In both of the other assemblies the austenitic pipe was increased in thickness to match the ferritic pipe thickness.

3. The Babcock & Wilcox test specimen was cooled to room temperature during each cycle. The Public Service test specimens were cooled to not less than 200 F. and the Navy quench tests were carried down only to the saturated steam temperature. This may be the important difference, as the maximum stress due to differential contraction occurs at the lower end of the cycle if we assume that the joint stress relieves itself at the operating temperature. Also, the Babcock & Wilcox test piece was held at the operating temperature for a whole week before cooling, allowing it to stress relieve itself to a larger extent, while in both of the other tests the time at operating temperature was relatively short with less opportunity for stress relief.

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[1] Mechanical Engineer, Engineering Dept., Public Service Electric and Gas Co., Newark, N. J.

[2] *Transactions*, Am. Soc. Mechanical Engrs., Vol. 71, No. 6, August, 1950. (*Paper No. 48–A–87*).

[3] *Transactions*, Am. Soc. Mechanial Engrs., Vol. 72, No. 7, October. 1950. (*Paper No. 50–S–23*).

It is of interest to note that in connection with the Kelcaloy joint in the Public Service test an incipient crack was found on the inner surface of the pipe where the two metals join, after about half of the test had been completed. This crack was very similar to that shown in the top photomicrograph of Fig. 11 of the Blaser paper. The crack, however, did not progress during the remainder of the test including the quench runs and this may be an indication of the value of this type of joint over the straight butt weld. It is noted that weld preparation "D" in Fig. 4 of the Carpenter paper withstood more cycling than any of the other types of weld grooves. This weld detail closely approaches the lap type of joint of the Kelcaloy transition piece.

Kelcaloy transition joints are being used on all four units at Sewaren Station. A careful inspection of the piping of the first unit after 15 months of operation, the major portion of which was at 1050 F., showed no indication of failure. Incidentally, a number of cracks were found in other parts of the austenitic piping system, all of which it is believed can be traced either to high stresses due to abrupt changes in section or defects in the base material or welds which were not detected during the assembly of the piping system.

It has been recognized by those who have given much thought to the problem that failure of welds between dissimilar alloys, particularly in heavy wall piping, is inevitable if the range and frequency of temperature changes in operation are sufficiently great. This work sponsored by the Babcock & Wilcox Co. adds considerably to the meager quantitative information on the subject and will be invaluable in many industrial installations where both ferritic and austenitic materials are involved.

MR. N. L. MOCHEL.[4]—Relative to the joining together of dissimilar materials, reference has already been made by Mr. Weisberg to the use at Sewaren, N. J., where six are already in service and two more will be installed next year. These are rather large and heavy sections where the main steam line is joined through the throttle valve of the turbine.

Also for turbine construction for service at 1050 F., it has been found expedient to join together dissimilar materials. In one case 1 Cr-½ Mo has been joined to 18 Cr-8 Ni Cb, and in another case 2 Cr-1 Mo to 18 Cr-8 Ni Cb.

Mr. Weisberg mentioned the fact that some joints have been examined after 15 months of service with a steam temperature of 1050 F. In the installation where the incoming 18 Cr-8 Ni Cb steam piping has been joined to the outer casing of the turbine, some six joints are involved between the austenitic material and the 1 Cr-½ Mo outer turbine casing. These welds were very carefully examined after 15 months of service and were found to be entirely satisfactory and free of any trouble. Of course, the temperature is somewhat lower than the 1100 F. mentioned by Mr. Carpenter.

It was not possible to examine the internal welds where the steam piping enters the nozzle chamber and where the nozzle chamber and the 18 Cr-8 Ni Cb Mo steel casting is joined to the inner turbine casing of 2 Cr-1Mo. However, in all work of this sort, we have been very careful to do one of the things that Mr. Carpenter mentioned at the very end of his discussion. We believe in boring out the joint and turning it outside to a uniform section. The backing up strip and the full effects of backing up strip are removed, so that smooth machined interior and exterior surfaces are obtained.

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[4] Manager, Metallurgical Engineering, Westinghouse Electric Corp., Philadelphia, Pa.

We have also seen fit to join together some smaller piping where drain lines of ferritic material might be joined to nipples or other devices for joining to the heavy throttle valves or turbine casings, as the case may be. The same practice is followed here as in the large piping. The inside of the joint is bored out and the outside is turned so that a perfectly smooth contour both inside and out is obtained at these points.

Also, I suspect that the stresses in the case of these joints I have mentioned are considerably lower, and temperatures certainly are lower, than the majority of those referred to by Mr. Carpenter.

MR. O. R. CARPENTER (*author's closure*).—I wish to repeat one point which may not have been clearly enough brought out. This had to do with the operating temperature and its relation to the failure described. The work reported by this paper and that of Blaser, Eberle, and Tucker shows 1100 F. or more to be the critical range. Indications are that joints operating at lower temperatures, say 1050 F., may be very satisfactory under constant conditions and over a long operating period.

The purpose of the work reported in this paper was not to predict the time of failure nor the critical operating temperature of a dissimilar metal joint. It has been to show that under certain conditions this type weld will fail. These conditions have been established in the paper and certain precautions for avoiding them recommended.

MESSRS. R. U. BLASER, F. EBERLE, AND J. T. TUCKER, JR. (*authors' closure*).—We thank Messrs. Weisberg and Mochel for their comments. It is quite evident that present knowledge of dissimilar weld joint properties is inadequate, and for this reason opinions may differ regarding some aspects of this problem.

Take, for example, the questions raised by Mr. Weisberg. To us, the advantages of a postweld treatment appear to be doubtful since residual welding and thermal expansion stresses exceeding the operating creep strength of the material will be reduced to the same level by plastic creep after the joint has been in operation for some time.

With respect to Mr. Weisberg's comments regarding the weld joint design of our test specimen, we wish to point out that cracking started on the outside of the pipe. The greatest stress concentration, however, is on the inside at the change in cross-section and backing ring location. If such stress concentrations are an important factor, cracking should have started at this inside location.

We agree that our extending the cooling cycle to room temperature was undoubtedly responsible for a greater stress range, and it is quite possible that this factor in conjunction with the longer holding time at operating temperature constitutes the important difference between the Babcock & Wilcox test and Mr. Weisberg's test.

Inasmuch as an incipient crack occurred at the internal weld junction of the Kelcaloy joint in Mr. Weisberg's test which admittedly was less severe than the Babcock & Wilcox test, it would, indeed, be interesting to know what would have happened if the Kelcaloy joint had been subjected to these more severe conditions.

There is some comfort in Mr. Mochel's statement that there are several ferritic-austenitic weld joints which have been in service for 15 months without indications of trouble. However, we should like to point out that unless the number, severity, and type of the cycles of these joints are known, a comparison with the Babcock & Wilcox test is not conclusive.

HARDENING OF AUSTENITIC STAINLESS STEELS BY MECHANICAL WORKING AT SUB-ZERO TEMPERATURES*

By N. A. Ziegler[1] and P. H. Brace[2]

Synopsis

It has been discovered that austenitic stainless steels, having approximate compositions of 12 to 30 per cent chromium and 11 to 20 per cent nickel, can be made unusually hard by plastically deforming them at sub-zero temperatures and thus promoting the gamma to alpha transformation. Hardness values thus obtained can be increased still further by a subsequent aging heat treatment of about 750 F. (400 C.).

Samples of suitable compositions have thus been hardened all the way through by rolling or wire drawing. Other physical properties are affected accordingly, so that in an 18 per cent chromium −8 per cent nickel strip, rolled at −300 F. (−185 C.) and aged, tensile strength of 295,000 psi., yield stress of 290,000 psi., proportional limit of 264,000 psi., elongation of 2.0 per cent, reduction of area of 24.8 per cent and hardness of 607 V.P.N. (Vickers diamond pyramid number) have been obtained.

Exploratory tests indicate that the resistance to corrosion is not seriously impaired by the processing required to produce the exceptionally high mechanical properties noted above.

In connection with some fundamental investigations of austenitic stainless steels, a large variety of compositions, containing different amounts of chromium, nickel, molybdenum, and carbon, was tested for Charpy impact resistance at the temperature of liquid nitrogen (about −300 F. or −185 C.). In the course of that work it was observed that some of the steels of 18–8 or 18–8 with 2 per cent molybdenum types (non-magnetic at room temperature when rapidly cooled from 1900 to 2000 F. (1038 to 1093 C.), upon being broken at −300 F., became, adjacent to the fracture, magnetic to such an extent that when the pieces were at room temperature, they could be lifted by an Alnico magnet. However, even at a short distance away from the fracture these bars remained non-magnetic. Bars of the same steels broken at room temperature remained non-magnetic all over. Nor were these bars rendered magnetic by immersion in liquid nitrogen. Consequently, the only conclusion which could be reached from these observations was that the increase in magnetic permeability was caused by the occurrence of severe plastic deformation at the low temperature.

Increased magnetic permeability in austenitic stainless steels resulting from mechanical working performed at sub-zero temperatures has also been observed

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TABLE I.—EFFECT OF AGING ON THE HARDNESS OF AUSTENITIC STAINLESS STEELS.
Vickers Hardness No. V.P.N./30.[a]

		Nominal Composition, per cent				Chemical Analysis, per cent									Annealed and Aged			Mechanically Worked at Room Temperature and Aged			Kept at −300 F. and Aged			Mechanically Worked at −300 F. and Aged		
Sample No.	Heat No.	Chromium	Nickel	Carbon, Maximum	Molybdenum	Silicon	Manganese	Sulfur	Phosphorus	Carbon	Nickel	Chromium	Molybdenum	Nitrogen	"Annealed." Air Quenched from 1900 F. 1	"Annealed" and Aged 750 F. 24 hr. 2	"Annealed." Aged 750 F. 24 hr. and 1000 F. 24 hr. 3	Worked at Room Temperature 4	Worked at Room Temperature and Aged at 750 F. 24 hr. 5	Worked at Room Temperature, Aged at 750 F. 24 hr. and 1000 F. 24 hr. 6	Kept at −300 F. for 24 hr. 7	Kept at −300 F. 24 hr. and Aged at 750 F. for 24 hr. 8	Kept at −300 F. 24 hr. Aged at 750 F. 24 hr. and at 1000 F. for 24 hr. 9	Worked at −300 F. 10	Worked at −300 F. and Aged at 750 F. for 24 hr. 11	Worked at −300 F. and Aged at 750 F. for 24 hr. and at 1000 F. for 24 hr. 12
1	3334	15	8	0.1	0	0.70	0.78	0.010	0.022	0.06	7.87	14.77	0.03	.....	243	253	287	406	393	326	233	270	267	428	424	314
2	4339	18	8	0.1	0	0.89	0.63	0.014	0.028	0.08	8.12	17.84	0.04	.....	133	223	189	439	451	337	223	228	191	453	559	268
3	4341	21	8	0.1	0	0.84	0.71	0.011	0.028	0.07	7.99	20.57	Tr.	.....	181	189	168	371	441	309	237	277	167	371	512	249
4	4467	15	14	0.1	0	0.61	0.52	0.017	0.026	0.05	14.01	15.41	0.01	.....	155	140	125	253	254	257	142	137	123	235	315	241
5	4448	18	14	0.1	0	0.65	0.52	0.016	0.014	0.06	14.61	17.74	0.14	.....	145	135	139	231	232	224	142	208	135	214	212	189
6	4466	21	14	0.1	0	1.31	0.56	0.016	0.028	0.06	13.09	20.90	0.01	.....	150	140	127	250	308	275	146	145	133	234	294	257
7	4151	15	8	0.1	2	1.00	1.15	0.009	0.030	0.05	8.00	14.88	2.19	0.048	177	167	169	355	366	364	168	200	167	398	524	337
8	4152	18	8	0.1	2	1.00	1.09	0.015	0.028	0.08	8.00	18.28	2.24	0.040	178	197	195	317	363	249	194	222	190	450	596	324
9	4173	21	8	0.1	2	0.77	0.83	0.018	0.030	0.08	8.25	21.08	2.40	0.037	183	239	175	317	444	278	261	221	211	412	595	311
10	4184	15	14	0.1	2	0.72	0.80	0.021	0.030	0.06	13.46	15.43	2.20	0.029	159	171	141	210	252	240	132	158	132	266	377	281
11	4185	18	14	0.1	2	0.84	0.86	0.022	0.024	0.06	13.52	18.18	1.86	0.029	173	146	130	262	272	236	176	165	144	280	325	190
12	4186	21	14	0.1	2	0.72	0.82	0.021	0.022	0.05	13.52	21.61	2.12	0.026	187	204	186	255	285	281	201	202	171	265	305	197

[a] Each figure is an average of two or more readings.

by Mathieu.[3] However, it appears that he did not go beyond the recording of his observations. Obviously (as also pointed out by Mathieu), this change in permeability must have been caused by the increases in the amount of ferrite that accompany the transformation from gamma to alpha state, which is promoted by the combination of plastic deformation and low temperature. At the same time, if such is the case, one might expect that the transformation would also be associated with a change in hardness and other physical properties. A few Vickers hardness tests taken on the Charpy bars broken at −300 F. confirmed this opinion: adjacent to the fracture the hardness had, at least in some cases, increased to well over 400 V.P.N. In the bars of the same compositions, broken at room temperature, hardness adjacent to the fracture also increased but to lesser values than those observed in the bars broken at sub-zero temperatures.

It appears that this phenomenon is not unlike that observed in Hadfield manganese steels, which, after quenching from a high temperature, are predominantly austenitic, non-magnetic and relatively soft, but upon subsequent plastic deformation at room-temperature become ferritic, magnetic, and show increased hardness. The main difference is that in the Hadfield steels this phenomenon occurs at room temperature while in the case of chromium-nickel stainless steels it takes place at sub-zero temperatures.

TESTS ON BROKEN CHARPY IMPACT BARS

After establishing the fundamental facts noted above, a broader survey of broken Charpy bars was undertaken. For those preliminary studies, they were considered as a convenient working material because each one of them contained a severely deformed portion, adjacent to the fracture, that merged into the undisturbed "annealed" metal in the shanks of the bars.

The nominal compositions and the chemical analyses of the selected cast steel samples are given in Table I. It should be noted that all are low in carbon (under 0.1 per cent) and that they fall into two main groups, one containing no molybdenum and the other about 2 per cent molybdenum. Each group is divided into two sub-groups, one with about 8 per cent nickel and the other 14 per cent nickel. In each of the sub-groups are three steels whose respective chromium contents are about 15, 18, and 21 per cent, with other elements the same in all three.

Each steel was represented by two samples, one broken at room temperature and the other at −300 F. In other words, each composition was studied in four conditions (Table I, columns 1, 4, 7, and 10):

(*a*) "Annealed," that is air cooled from 1900 to 2000 F. (1038 to 1093 C.),

(*b*) "Annealed," and plastically deformed at room temperature,

(*c*) "Annealed," and chilled in liquid nitrogen (−300 F.), and

(*d*) "Annealed," and plastically deformed at −300 F.

The question was then raised as what would be the maximum temperature at which hardness obtained by the sub-zero plastic deformation would persist and at what temperature softening would commence. The answer was sought by aging all of the above samples at progressively higher temperatures and measuring hardness after each aging step. In Table I the hardnesses obtained after 750 F. (400 C.) aging are given in columns 2, 5, 8, and 11, and after 1000 F.

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[3] K. Mathieu, "Einfluss von Legierungsgehalt und Versuchsbedingungen auf die Gamma-Alpha Umwandlung bei der kaltverformung austenitischer Stahle," *Archiv fur das Eisenhüttenwesen*, Vol. 16, No. 6, December, 1942, p. 215.

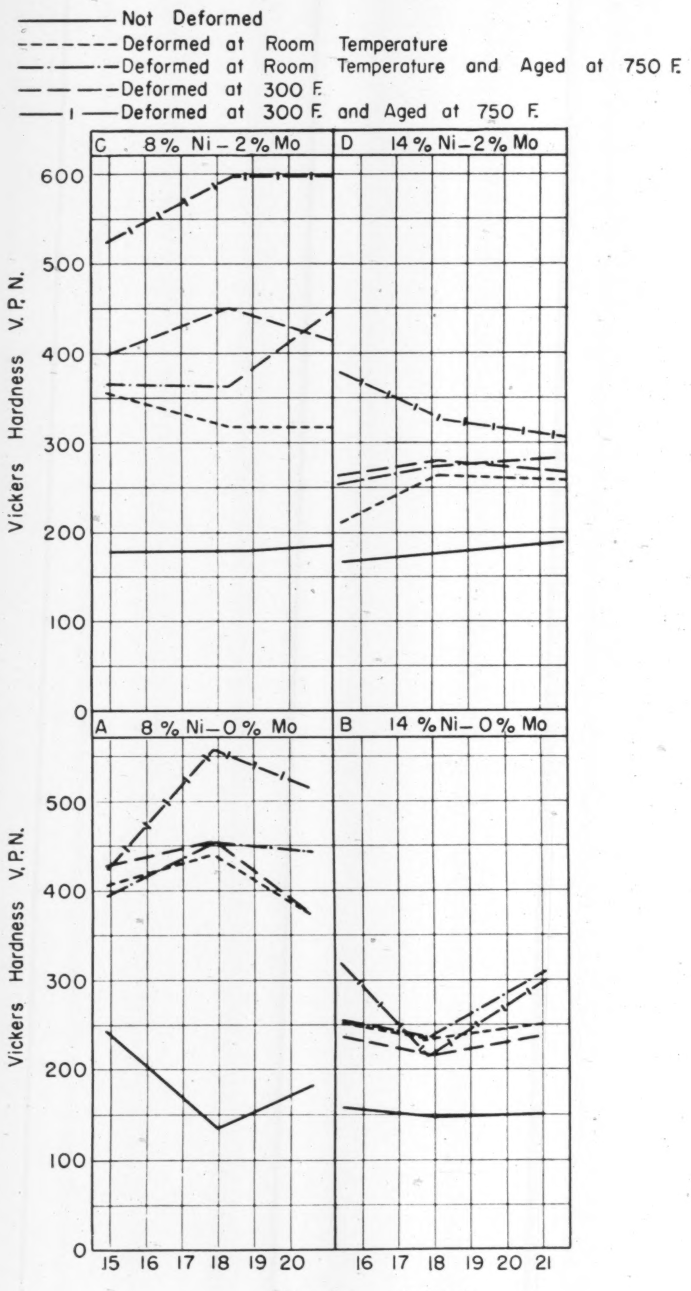

FIG. 1.—Effect of Chromium on the Hardening of Plastically Deformed Austenitic Steels.

(538 C.) aging—in columns 3, 6, 9, and 12.[4]

In all the steels the hardness values produced by plastic deformation were maintained or increased by aging at temperatures up to 750 F. At higher temperatures softening commenced and at about 1000 F. progressed fairly rapidly. A mere chilling in liquid nitrogen had only a slight (if any) hardening effect, as compared to the "annealed" condition.

Figure 1 is a graphic representation of the effect of chromium on hardness within the aforementioned four subgroups (8 per cent nickel, 0 per cent molybdenum; 14 per cent nickel, 0 per cent molybdenum; 8 per cent nickel, 2 per cent molybdenum; and 14 per cent nickel, 2 per cent molybdenum) in the following five physical "conditions":

(*a*) "Annealed," that is rapidly cooled after heating to 1900 to 2000 F.,

(*b*) "Annealed," then plastically deformed at room temperature,

(*c*) "Annealed," then plastically deformed at room temperature and aged at 750 F.,

(*d*) "Annealed," then plastically deformed at −300 F., and

(*e*) "Annealed," then plastically deformed at −300 F. and aged at 750 F.

It will be seen that for a given steel, the hardness of the samples plastically deformed at sub-zero temperatures

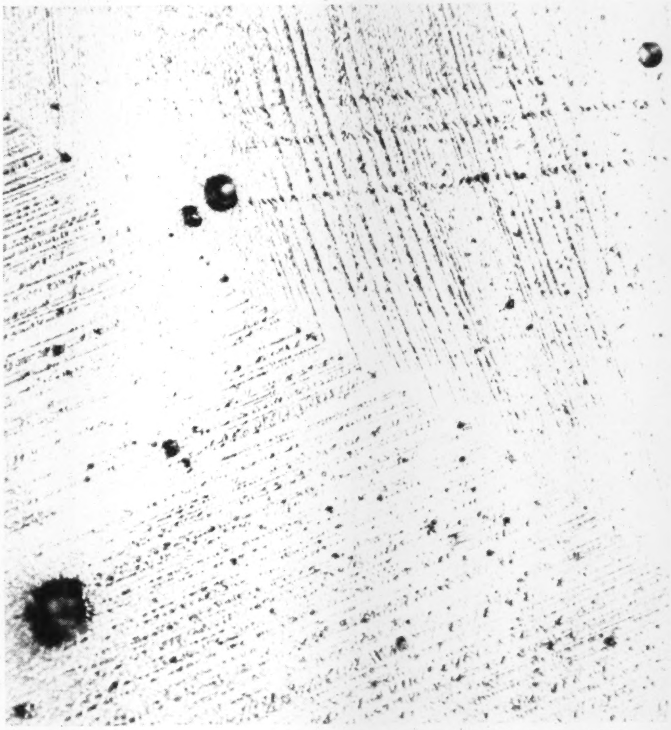

FIG. 2.—Microstructure of Sample No. 4 (Table I) (× 500).
Worked at −300 F. Aged at 750 F. Etched in *aqua regia*.

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[4] The effects of aging steps at intermediate temperatures are omitted.

TABLE II.—EFFECT OF AGING ON HARDNESS OF AUSTENITIC STAINLESS STEELS.
Vickers Hardness No. V.P.N./10.[a]

		Nominal Composition, per cent				Chemical Analysis, per cent										Shot Peened at Room Temperature and Aged			Shot Peened at −300 F. and Aged			Forged at Room Temperature and Aged			Forged at −300 F. and Aged		
Sample No.	Heat No.	Chromium	Nickel	Carbon, Maximum	Molybdenum	Silicon	Manganese	Sulfur	Phosphorus	Carbon	Nickel	Chromium	Molybdenum	Nitrogen	"Annealed" Air Quenched 1900 F.	Peened at Room Temperature	Peened at Room Temperature and Aged at 750 F. 24 hr.	Peened at Room Temperature, Aged at 750 F. 24 hr. and 1000 F. 24 hr.	Peened at −300 F.	Peened at −300 F. and Aged at 750 F. 24 hr.	Peened at −300 F. Aged at 750 F. 24 hr. and 1000 F. 24 hr.	Forged at Room Temperature	Forged at Room Temperature. and Aged at 750 F. 24 hr.	Forged at Room Temperature, Aged at 750 F. 24 hr. and 1000 F. 24 hr.	Forged at −300 F.	Forged at −300 F. and Aged at 750 F. 24 hr.	Forged at −300 F., Aged at 750 F. 24 hr. and 1000 F. 24 hr.
13[b]	4339	18	8	0.1	0	0.89	0.63	0.014	0.028	0.08	8.12	17.84	0.04	.....	180	...	...	...	471	545	373	...	...	...	410	443	333
14[b]	4152	18	8	0.1	2	1.00	1.09	0.015	0.028	0.08	8.00	18.28	2.24	0.040	202	293	375	339	448	553	417	382	390	346	369	445	315
15[c]	5143	18	8	0.1	0	0.42	0.39	0.012	0.010	0.07	8.18	17.80	....	.....	184	442	441	327	462	522	306	349	358	313	462	513	349

[a] Each figure is an average of two or more readings.
[b] Cast.
[c] Rolled.

reached higher values than did the hardness of the corresponding specimens deformed at room temperature. In some cases (Fig. 1) this difference is quite substantial. Likewise, subsequent aging at 750 F. had a greater effect on the former than on the latter. In the two sub-groups containing 8 per cent nickel (Fig. 1 (*A*) and (*C*)) the effects of sub-zero deformation and of subsequent aging are much more pronounced than in the other two, containing 14 per cent nickel (Fig. 1 (*B*) and (*D*)). The difference is, no doubt, due to the austenite-stabilizing effect of nickel. In the former groups (8 per cent nickel and 8 per cent nickel, 2 per cent molybdenum) the hardness of some of the sub-zero-deformed-and-aged samples was well over 500 V.P.N.

Figure 2 is a typical microstructure of one of the aforementioned samples, plastically deformed at −300 F. and aged at 750 F. It shows a junction of two initially austenitic grains and the etching pattern resulting from the precipitate formed on the slip-planes under the stimulus of sub-zero deformation and subsequent aging heat treatment.

PLASTIC DEFORMATION BY SHOT PEENING AND FORGING

Three 18–8 compositions were selected for the investigation of the effect of plastic deformation by shot-blast peening and forging. Two of them were as cast and one hot rolled (Table II). They were "annealed" by air cooling from 1900 to 2000 F., and then individual samples were subjected to:

(*a*) Shot Peening at room temperature,[5]

(*b*) Shot Peening at −300 F.,

(*c*) Forging at room temperature,[5] and

(*d*) Forging at −300 F.

Although no attempts were made to measure the amount of cold deformation, it may be mentioned that reduction by forging was carried to about one-third of the original thickness, while shot peening was performed over a period of 1 min.

It should be understood that plastic deformation by shot peening produced only a superficial skin effect. However, this plastically deformed skin was sufficiently thick to be tested for Vickers hardness, using a light load.

Hardness measurements on each sample were taken:

(*a*) As plastically deformed,

(*b*) Aged at 750 F. and

(*c*) Aged at 1000 F.

The data thus accumulated are recorded in Table II. It may be observed that shot peening produced higher hardness values than forging which, no doubt, was due to the more intense, though localized, plastic deformation produced by shot peening. Likewise, sub-zero plastic deformation in all cases had a greater effect than that imposed at room temperature. Aging at 750 F. produced a far greater increment in hardness in the sub-zero deformed samples, than in those deformed at room temperature. Moreover, this effect is more pronounced in the shot-peened than in the forged samples. Aging at 1000 F. softened all of them.

Figures 3 and 4 represent microstructures and micro-hardness studies at the surfaces of samples Nos. 13 and 14 (Table II), respectively, after shot peening at −300 F. and aging at 750 F. In both cases, a predominantly austenitic structure with some ferrite (frequently present in cast structures of steels of this type) can be observed. Precipitation in the cleavage planes of the parent-austenite, produced by the low-temperature plastic deformation and subsequent aging, can readily be seen, in both cases. Micro-hardness distribution

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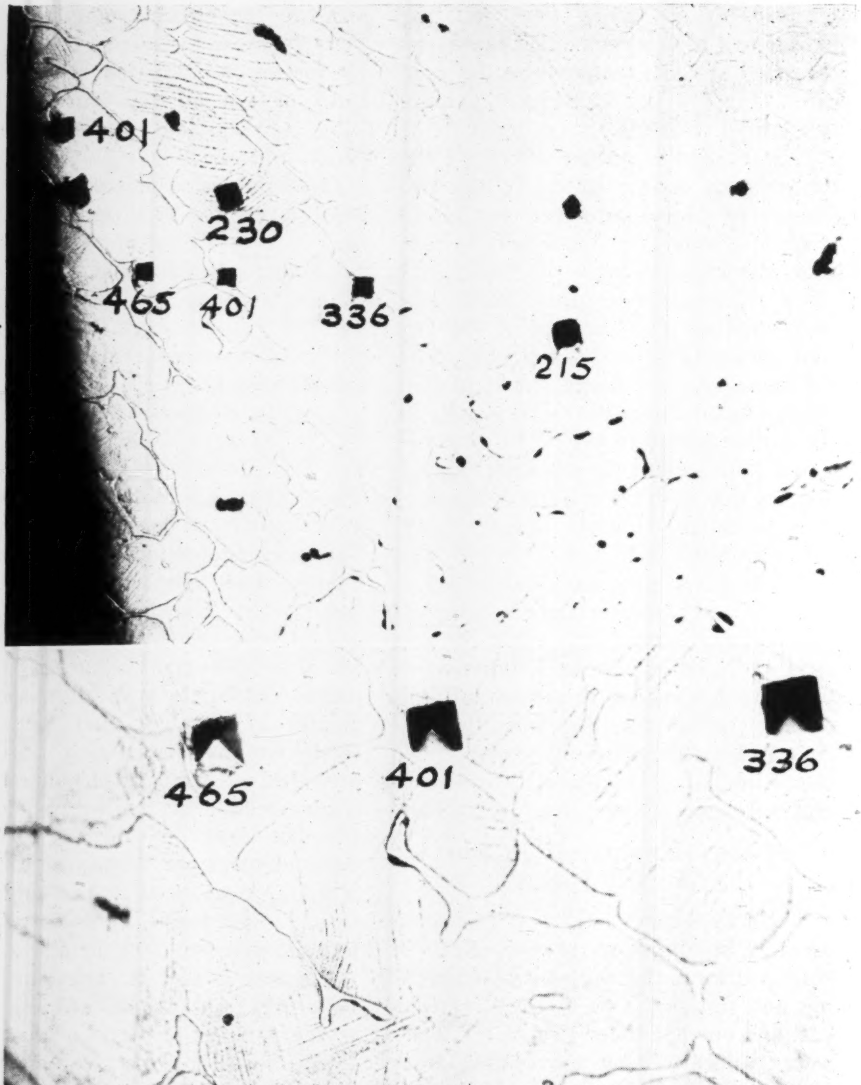
[5] Sample No. 13 was not worked at room temperature.


401
230
465
401
336
215
465
401
336

(a) × 150.

(b) × 400

FIG. 3.—Microstructure of Sample No. 13 (Table II).
Shot Peened at −300 F. Aged at 750 F. Etched in *aqua regia*.

studies indicate that the hardness at the surface is rather high and that it gradually diminishes towards the inside of the sample. Micro-hardness values measured at the surface (Figs. 3 and 4) are lower than the corresponding hardness figures recorded in Table II. This probably is due to the fact that it was difficult to obtain symmetrical micro-hardness impressions at the extreme edge of the sample.

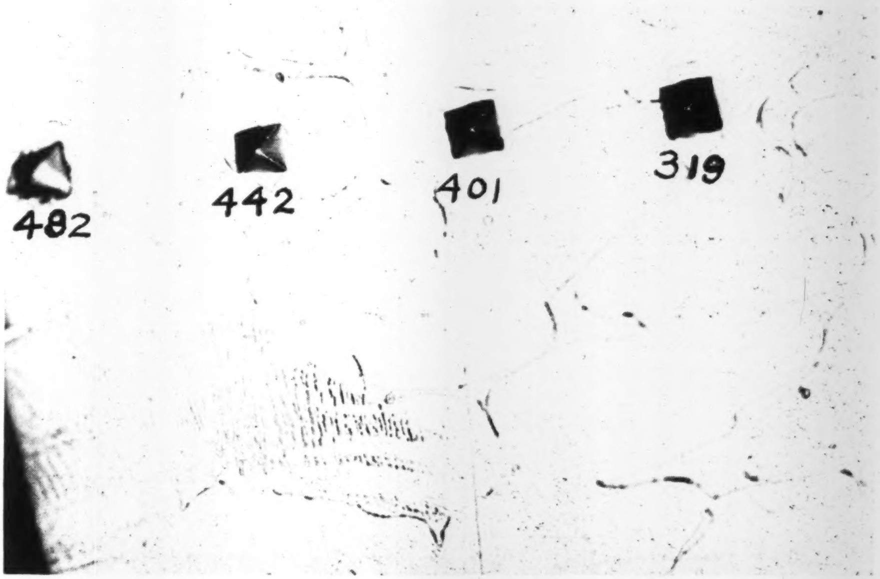


FIG. 4.—Microstructure of Sample No. 14 (Table II) (× 450).
Shot Peened at −300 F. Aged at 750 F. Etched in *aqua regia*.

MECHANICAL WORKING BY ROLLING AND WIRE DRAWING

The next phase of this investigation was a study of the effects of sub-zero mechanical working,[6] as obtained by rolling and wire drawing, as well as the effect of the subsequent aging. Table III represents nominal compositions and chemical analyses, mechanical working conditions, and the resultant hardness values for representative specimens of the alloys used.

Excepting sample No. 27, which was worked by wire drawing, all of the samples were worked by rolling. Sample Nos. 16 to 19 were commercial steels of 18-8 type, rolled in lengths of several feet so that, besides hardness, their other properties, to be discussed later on, could be investigated. Sample Nos. 20 to 26 were small samples cut off from cast coupon blocks and rolled out to lengths of a few inches. From the previous experiments with Charpy bars it was known that the chemical compositions of the latter group were such that they would be "border-line" cases. The main object of testing them was to determine the compositional range within which the alloys would be responsive to sub-zero working.

Each sample was worked down to the same percentage reduction either at room temperature (about 77 F.

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[6] "Plastic deformation" is distinguished from "mechanical working" by the fact that in the latter case the amount of plastic deformation was predetermined and measured.

TABLE III.—MECHANICAL WORKING BY ROLLING AND WIRE DRAWING.

Sample No.	Heat No.	Nominal Composition, per cent				Chemical Analysis, per cent									Nominal Working Temperature, deg. Fahr.	Thickness, in.				Number of Passes	Total Reduction of Thickness, per cent	Hardness, V.P.N.[d]			Remarks
		Chromium	Nickel	Molybdenum	Columbium	Silicon	Manganese	Sulfur	Phosphorus	Carbon	Chromium	Nickel	Molybdenum	Columbium		Initial		Final				As Received	As Worked	As Worked and Aged at 750 F.	
16		18	12	0	0	Normal Percentages					18.76	11.13	0.01	0	77^a	1/16	As Received	1/64	Approximate	9	75.0	170 to 180	376	418	Commercial Rolled Steels Worked by Rolling.
															-300^b	1/16		1/64		9	75.0		493	585	
17		18	12	0	0	Normal Percentages					18.76	11.13	0.01	0	77^a	1/16		1/64		13	75.0	170 to 180	360	410	
															-300^b	1/16		1/64		13	75.0		499	602	
18		18	9	0.2	0	Not Analyzed									77^a	1/16		1/64		13	75.0	170 to 180	398	480	
															-300^b	1/16		1/64		13	75.0		579	660	
19		17–19	8–10	. . .	.	Not Analyzed									77^a	3/16		3/64		13	75.0	170 to 180	367	437	
															-300^b	3/16		3/64		13	75.0		517	625	
20	5822	21	20	0	0	0.63	0.58	0.012	0.030	0.03	20.96	19.64	0.01	0	77^a	0.127	Machined to Thickness	0.032	Measured	13	74.8	127	366	426	Cast Steels. "Border-line" Compositions. Worked by Rolling.
															-300^b	0.126		0.033		14	73.8	127	407	455	
21	5834	24	20	0	0	0.70	0.74	0.014	0.030	0.03	24.08	21.02	0.01	0	77^a	0.125		0.032		11	74.4	126	359	391	
															-300^b	0.125		0.032		12	74.4	126	384	439	
22	4645	18	4	0	0	0.63	0.57	0.010	0.012	0.07	17.17	4.49	0.01	0	77^a	0.124		0.031		13	75.0	331	440	501	
															-300^b	0.125		0.031		12	75.2	348	487	528	
23	4679	18	4	2	0	0.72	0.76	0.007	0.020	0.05	18.19	4.44	2.28	0	77^b	0.126		0.031		12	75.3	218	453	535	
															-300^b	0.125		0.032		12	74.3	223	476	540	
24	5791	12	20	0	0	0.42	0.51	0.024	0.016	0.04	11.42	19.64	0.01	0	77^a	0.125		0.031		11	75.1	109	313	354	
															-300^b	0.125		0.032		12	73.6	111	372	405	
25	5931	12	20	0	1	0.49	0.70	0.012	0.022	0.09	12.26	20.16	0.02	0.97	77^a	0.125		0.031		12	75.2	141	334	375	
															-300^b	0.124		0.031		13	75.0	140	381	429	
26	5844	30	20	0	0	1.05	0.78	0.010	0.028	0.01	30.21	19.45	0.01	0	77^a	0.126		0.030		11	76.2	203	361	407	
															-300^b	0.123		0.032		13	74.0	205	365	438	
27	5797	18	8	0	0	Not Analyzed									77^a	0.057	Diam.	0.040		5	[c]50.8	454	482	547	Commercial Wire, Further Worked by Wire-drawing.
															-300^b	0.057				5	[c]50.8	454	540	641	

[a] Room Temperature.
[b] Liquid Nitrogen (−320 F. or −195 C.) Used as Coolant.
[c] Reduction of Cross-Section.
[d] Each hardness value is an average of several readings.

(25 C.)) or at about −300 F. The latter was done by chilling the specimens in liquid nitrogen, passing them through the rolls, immediately putting them back in liquid nitrogen, and repeating this operation until the desired reduction was obtained.

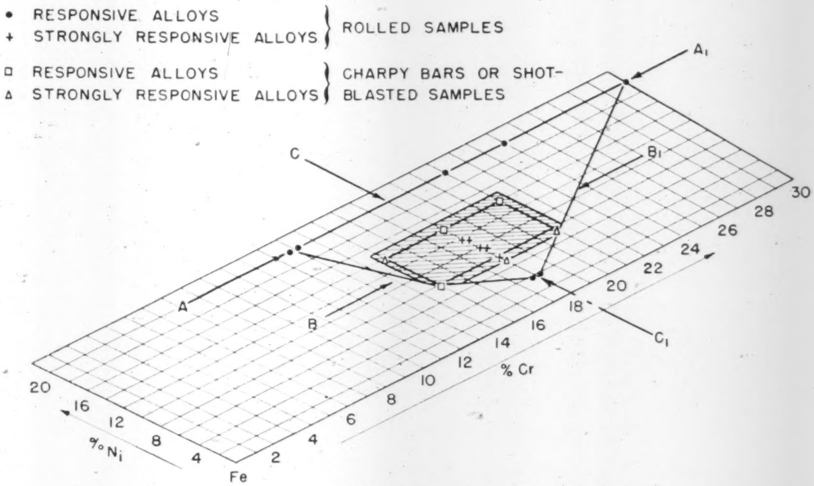
● RESPONSIVE ALLOYS
+ STRONGLY RESPONSIVE ALLOYS
ROLLED SAMPLES
□ RESPONSIVE ALLOYS
△ STRONGLY RESPONSIVE ALLOYS
CHARPY BARS OR SHOT-BLASTED SAMPLES
A
A₁
B
B₁
C
C₁
2
4
6
8
10
12
14
16
18
20
22
24
26
28
30
% Cr
20
16
12
8
4
% Ni
Fe

FIG. 5.—Composition Range of the Experimental Alloys.

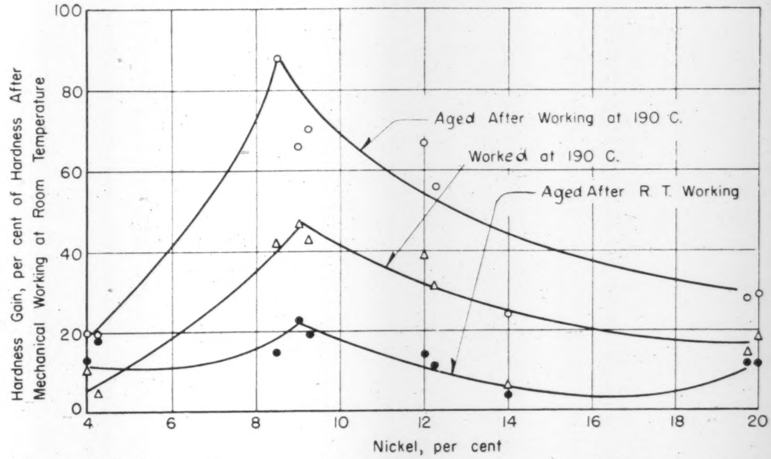

FIG. 6.—Effect of Nickel on Hardening of Steels Containing 18 per cent Chromium (Cross-Section C-C₁, Fig. 5).

With the exception of the sample No. 27, all the specimens were given a preparatory heat treatment by holding them at about 1900 F. for approximately 30 min. and quenching them in oil (sample Nos. 16 to 19), or in water (sample Nos. 20 to 26). Sample No. 27 was not given any preparatory heat treatments and mechanical working was effected by drawing it through a series of successively smaller dies so that its cross-sectional area was reduced by approximately 50 per cent in five passes. Several feet of wire were thus drawn in order to provide enough material to perform, besides hardness tests, some other experiments to be discussed presently.

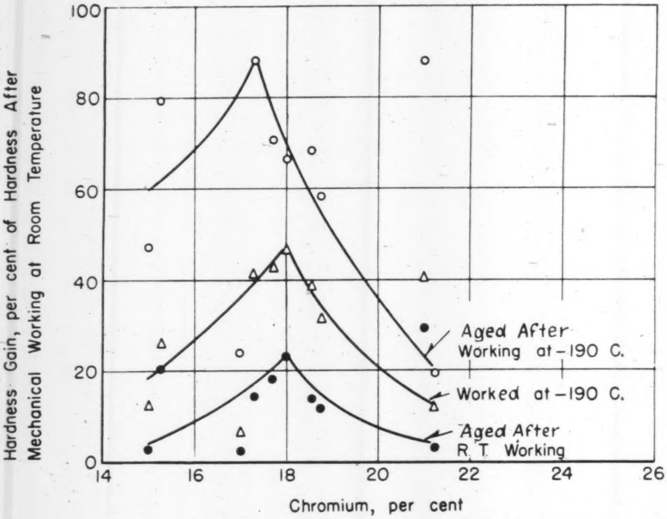

FIG. 7.—Effect of Chromium on Hardening of Steels Containing 8 to 14 per cent Nickel (Cross-Section B-B$_1$, Fig. 5).

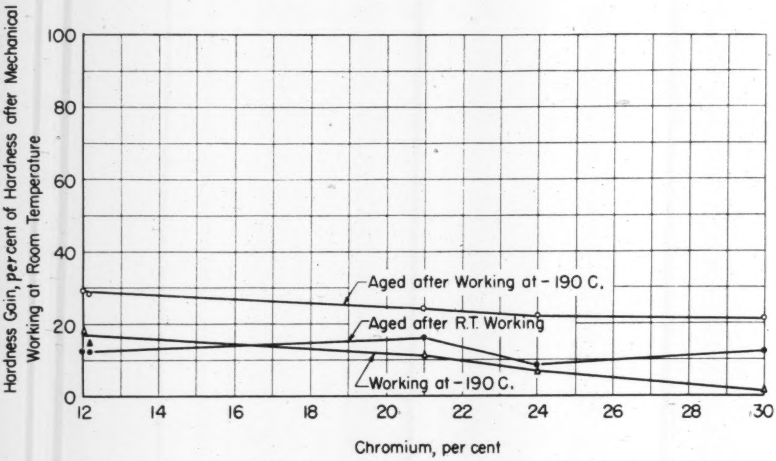

FIG. 8.—Effect of Chromium on Hardening of Steels Containing 20 per cent Nickel (Cross-Section A-A$_1$, Fig. 5).

After completing the mechanical working operations, two specimens were taken from each sample, one to represent the "as-worked" condition, while the other was aged for about 4 hr. at 750 F. and air cooled. Hardness measurements were made on all of them.

Mechanical working, at whatever temperature, produced in all of the samples

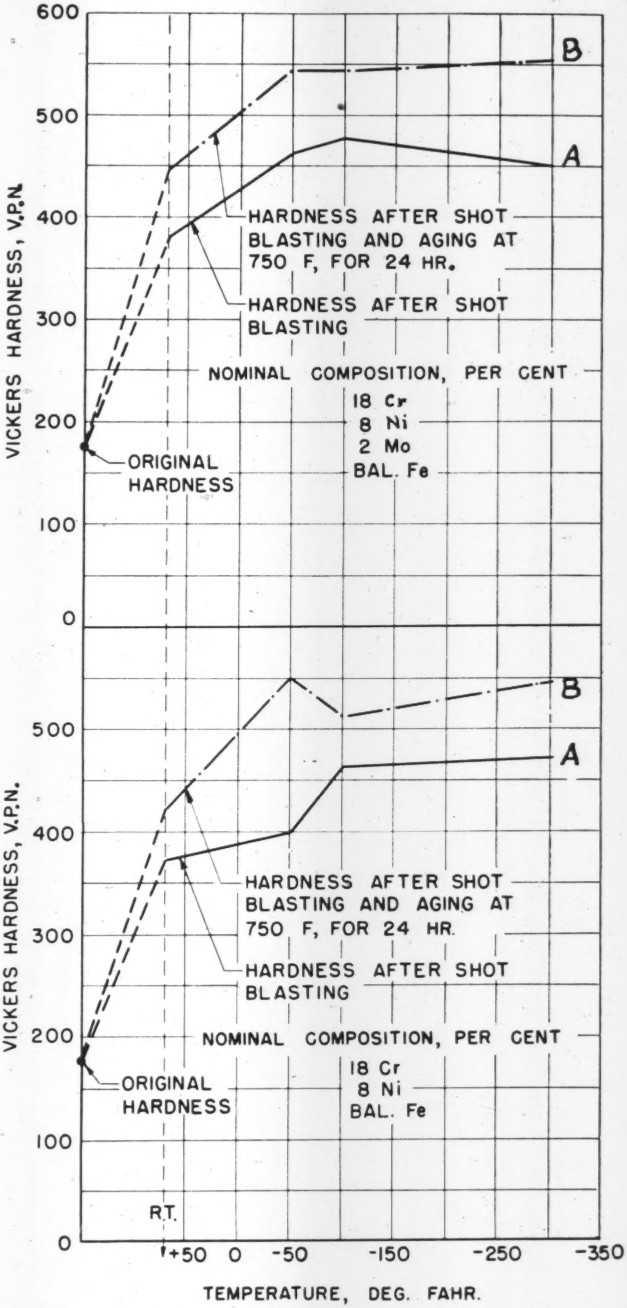

FIG. 9.—Effect of (A) Plastic Deformation at Various Temperatures and (B) Plastic Deformation at Various Temperatures Followed by Aging at 750 F. for 24 hr. on Hardness of Austenitic Steels.

a marked increase in hardness. Aging heat treatment caused further measurable increases. However, even without aging, the gains in hardness that resulted from the simultaneous application of sub-zero temperature and mechanical working were relatively great as compared with the gains resulting from mechanical working only at room temperature. Finally, when both mechanical working at sub-zero temperatures and aging heat treatment were applied, the maximum gains were obtained.

Figure 5 is a graphic representation on triaxial coordinates of the compositional range within which were found chromium-nickel steels that responded to sub-zero working and subsequent aging heat treatment, as noted in Tables I, II, and III. The shaded area represents a narrower field of compositions whose response to those treatments was particularly strong.

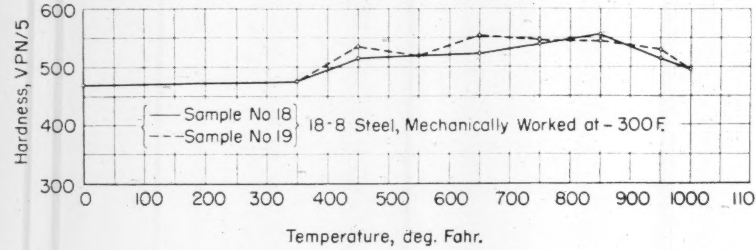

FIG. 10.—Effect of Aging for 1 hr. at Various Temperatures on Vickers Hardness.

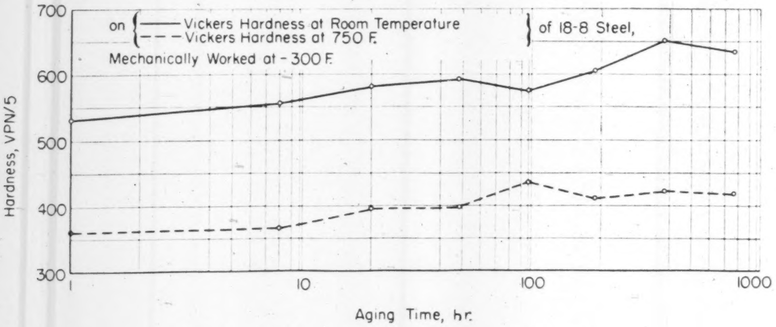

FIG. 11.—Effect of Time of Aging at 750 F.

Figure 6 represents the magnitude of the response for compositions taken along the cross-section C-C_1 of Fig. 5, that is along the nickel ordinate, chromium being constant at about 18 per cent. It might be noted that the responses of compositions either low (of the order of 4 per cent) or high (of the order of 20 per cent) in nickel are relatively small, but nevertheless significant. However, the responses of compositions containing about 7 to 14 per cent nickel are particularly high.

Figure 7 portrays the extent of the sub-zero hardening effect for compositions taken along the cross-section B-B_1 of Fig. 5, that is along the chromium ordinate for nickel contents between about 8 and 14 per cent. This group of alloys registered exceptionally high responses to sub-zero plastic working throughout the entire compositional range.

Finally, in Fig. 8 is shown the response for compositions taken along the cross-section A-A_1 of Fig. 5, that is along the chromium ordinate, nickel being constant at about 20 per cent. In this group of alloys, the responses are not very great, but nevertheless appreciable.

EFFECT OF THE TEMPERATURE OF PLASTIC DEFORMATION, OR WORKING AND AGING TIME AND TEMPERATURE

Figure 9 is a graphic representation of the effect of shot blasting at progressively lower temperatures, from room temperature down to that of liquid nitrogen, on the surface hardness of two steels, namely, 18-8 and 18-8 with 2 per cent molybdenum, (A) as shot-blasted, and (B) as shot-blasted and aged at 750 F. It appears from this chart that plastic deformation by any means at temperatures lower than about −100 F. was not particularly advantageous for the amounts of deformation here applied. However, for practical reasons it is desirable to start mechanical working at the temperature of liquid nitrogen (about −300 F.) in order that the metal may remain as long as possible in the sub zero temperature range in which the hardening is most pronounced.

TABLE IV.—PHYSICAL PROPERTIES OF COMMERCIAL 18-8 (SAMPLE NO. 28).

Preparation	Tensile Properties					Hardness
Rolling Temperature, deg. Fahr.	Tensile Strength, psi.	Yield Strength, psi.	Proportional Limit, psi.	Elongation, per cent	Reduction of Area, per cent	V.P.N.
+77[a]	214 000	168 000	120 000	7.6	55.0	460
−100[a]	244 000	236 000	192 000	4.2	32.2	512
−300[a]	218 500[c]	185 000[c]	218 500[c]	...[c]	....[c]	522
+77[b]	215 000	200 000	152 000	5.1	47.4	504
−100[b]	273 000	258 000	223 000	3.5	30.3	558
−300[b]	295 000	290 000	264 000	2.0	24.8	607

[a] Rolled at various temperatures.

[b] Rolled at various temperatures and aged at 750 F. for 4 hr.

[c] Defective Tensile Specimen.

Figure 10 indicates the effect of aging temperature (for 1-hr. periods) on the hardness of two samples of commercial 18-8 austenitic steels (sample Nos. 18 and 19, Table III), rolled at −300 F. It is apparent that there is a general tendency for hardness to increase with increase of aging temperature between 450 and 850 F. (232 and 454 C.). At higher aging temperatures, softening commences and at about 1000 F. progresses quite rapidly.

Figure 11 represents the effect of time of aging at 750 F. on the hardness, (a) at room temperature and (b) at 750 F.[7] of a sample of commercial 18-8 steel (Serial No. 19, Table III) rolled at −300 F. Even after 100 hr. the hardness trend is still upward in both cases.

TENSION AND TORSION TESTING

A number of tension tests were made on 18-8 strip or wire rolled or drawn at sub-zero temperatures. Some specimens were tested "as-rolled" while others were aged. Invariably, there were observed considerable increases in tensile strength, yield stress and proportional limit, associated with some reduction of static ductility.

Table IV gives a representative set of test data obtained on commercial 18-8 strip. Initially this material was in the form of rolled bars 1 in. wide by ¼-in. thick and 4-in. long. These were first heat treated by water quenching from approximately 1900 F., to put them in the soft condition, and then reduced in thickness to approximately $\frac{1}{16}$-in. by 18 to 19 rolling passes. The rolling

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[7] "Hot hardness."

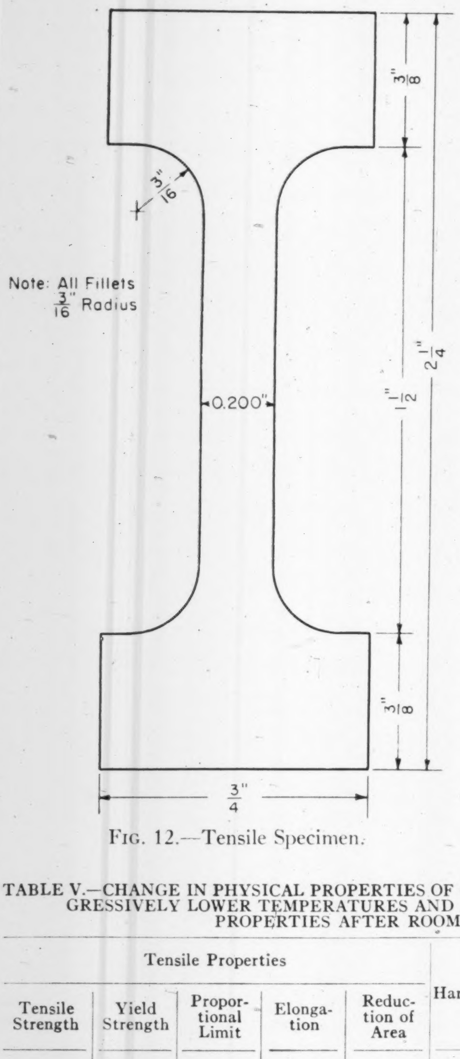

FIG. 12.—Tensile Specimen.

TABLE V.—CHANGE IN PHYSICAL PROPERTIES OF COMMERCIAL 18-8 SAMPLE NO. 28, ROLLED AT PROGRESSIVELY LOWER TEMPERATURES AND AGED AT 750 F. FOR 4 HR., AS PER CENT OF PROPERTIES AFTER ROOM TEMPERATURE ROLLING.

Tensile Properties					Hardness[a]	Rolling Temperature, deg. Fahr.	
Tensile Strength	Yield Strength	Proportional Limit	Elongation	Reduction of Area			
......	......	.....	....	....	......	+77	As Rolled
+14.0	+40.0	+60	−45	−42	+11.3	−100	
......	......	....	....	....	+13.5	−300	
+0.5	+19.0	+27	−33	−14	+ 9.6	+77	As Rolled and Aged at 750 F. for 4 hr.
+28	+54.0	+86	−54	−45	+21.3	−100	
+38	+73.0	+120	−74	−55	+32.0	−300	

[a] Hardness change in per cent of hardness after room-temperature rolling.

was done in three temperature ranges, namely, that of the room (77 F.), that obtained by cooling in dry ice (approximately −100 F.), and that obtained by immersion in liquid nitrogen (approximately −300 F.). Each of the rolled strips was cut into two parts, and one part of each piece was aged for 4 hr. at about 750 F. Hardness measurements and tension tests were made on both the "as-rolled" and "as-rolled-and-aged" specimens.

The shape and nominal dimensions of the specimens are shown by Fig. 12. Referring again to Table IV, it will be seen that the gain in hardness and tensile values caused by rolling only was greater for the two specimens rolled at sub-zero temperatures than for the one rolled at room temperature. Furthermore, the increments in hardness and tensile properties that resulted from aging were also greater for the specimens rolled at sub-zero temperatures than for that rolled at room temperature. Finally, the highest hardness and strength values were obtained in the specimens which were rolled at the lowest temperature (that is, that of liquid nitrogen).

Relative levels of tensile strength, yield stress and proportional limit fall into the same order as the corresponding hardness values. Ductility is lower for the harder and stronger specimens. Nevertheless, in the strongest one, a tensile strength of 295,000 psi., a yield stress of 290,000 psi., a proportional limit of 264,000 psi., and a hardness of 607 V.P.N. are associated with an elongation of 2 per cent and a reduction of area of 24.8 per cent.

The most significant figure, perhaps, is that for the proportional limit of the material rolled at −300 F. and aged, in comparison with the corresponding figure for material rolled at room temperature. The increase amounts to 120 per cent, that is more than double (Table V).

Another experiment showed that the torsional yield point of 18–8 wire (the same stock material as sample No. 27, Table III), in the form of a helical spring, was 156,000 psi. This represents a gain of about 46 per cent, relative to a yield stress of about 107,000 psi. for a similar spring formed from conventional cold drawn 18–8 wire.

WEAR TESTS

A series of wear tests was made, using as test materials several modifications of the 18–8 composition, mechanically worked or plastically deformed at about −300 F. by rolling, shot peening, forging, or burnishing. Specimens were tested in "as-worked" and "as-worked-and-aged" conditions.

The wear-testing machine used in this work imparts an oscillating rotary motion of approximately 5 deg. to one specimen[8] bearing upon a similar stationary specimen. Any desired contact pressure up to 4000 psi. can be applied. Evaluation of the wear characteristics of a material combination is made by observing the onset of leakage of air under controlled pressure between the lapped test surfaces and by visual inspection at the conclusion of the test. In the usual test procedure, frictional coefficients may be determined by measuring the degree of torsional flexure of the calibrated shaft holding the stationary specimen. Because there is no apparent relation between the coefficient of friction and seizure characteristics, frictional coefficients are not considered unless friction is excessive. In the present tests the friction was generally quite low.

Referring to the specimens used in these experiments, the rolled ones were prepared from strips similar in composition and processing to sample Nos. 16 to 19 (Table III). Rings, 1.7 in. outside diameter by 1.25 in. inside diameter, were cut from the original rolled strip. Some of them were used "as-rolled" while others were aged for 6 hr. at 750 F. These rings were mounted on a metal base, so that the assemblies had the dimensions of the standard wear test sample. The test surfaces were then ground and lapped.

The forged specimens were machined from solid blocks (previously chilled in liquid nitrogen and then sub-zero forged) to the wear test sample dimensions.

Shot peening and burnishing were performed at the temperature of liquid nitrogen on wear test specimens machined to the desired dimensions. In this group, the sub-zero plastic deformation produced only a superficial hardening effect. Since the surfaces thus produced were somewhat rough, they had to be slightly ground and lapped. Material thus removed from the surface, even though very slight in amount, constituted the most severely worked (that is the "best") part.

Among the three sub-zero-rolled-and-aged samples, one showed wear performance equal to or even better than the best of the wear-resistant metal combinations. This is quite remarkable because all austenitic stainless steels in any condition obtainable by conventional mechanical working and heat treatment, have notoriously low wear resistance and great tendency to galling under sliding contact. The other two samples, similarly treated and tested, showed a marked improvement as compared to the conventionally cold-worked austenitic stainless steels, but were greatly inferior to the first one. The only factor which, perhaps, might to some extent explain this difference in wear performance was that the "well-behaved" sample contained about 0.2 per cent carbon, while the other two had less than 0.1 per cent.

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[8] Standard specimen is a disk with a ring-shaped bearing surface, 1.7 in. outside diameter and 1.25 in. inside diameter.

Among the samples which were forged, shot peened or burnished at a sub-zero temperature, as well as those aged after the above treatments, some showed no and others only slight improvement as compared to the wear performance of conventionally cold-worked austenitic steels.

Hardness values for all of these samples, excepting the forged ones, were well over 500 V.P.N. Nevertheless with the exception of one, they all proved inferior to the standard wear-resistant materials, such as cobalt-base chromium alloys, even though the hardness of the latter frequently is below 500 V.P.N. As has been pointed out on numerous occasions, hardness is not the only property which determines a satisfactory wear resistance, and much more work must be done before the reasons for the inconsistencies can be made clear.

CORROSION TESTS

Some elementary corrosion tests were performed on several austenitic steels as (*a*) sub-zero worked and aged, and (*b*) annealed. Samples were immersed or partially immersed in a saturated sodium chloride solution. The test results were determined qualitatively by observing any possible water-line corrosion as well as discoloration of the metal and of the remaining liquid. On the strength of these tests, it might be concluded that all the worked and aged samples had about the same corrosion resistance as annealed 18–8 steel.

SUMMARY

1. It was discovered that austenitic chromium-nickel steels can be made unusually hard by plastically deforming them at sub-zero temperatures and thus promoting the gamma to alpha transformation.

2. This added hardness can be observed after mechanical working at about −50 F. (−46 C.) and progressively increases as the working temperature is lowered down to that of liquid nitrogen (about −300 F.).

3. Hardness values thus obtained can be increased still further by a subsequent aging heat treatment at about 750 F.

4. The compositional range within which this phenomenon was observed can be defined on the triaxial iron-nickel-chromium diagram by the following compositions: 18 per cent chromium, 4 per cent nickel; 12 per cent chromium, 20 per cent nickel; and 30 per cent chromium, 20 per cent nickel.

5. Within a narrower range defined by 15 per cent chromium, 8 per cent nickel; 15 per cent chromium, 14 per cent nickel; 21 per cent chromium, 14 per cent nickel; and 21 per cent chromium, 8 per cent nickel the response is particularly strong.

6. Within the latter range, hardnesses exceeding 500 V.P.N. can be obtained by sub-zero working alone. They can be further increased to over 600 V.P.N. by applying a subsequent aging heat treatment.

7. Samples of suitable compositions have thus been hardened all the way through by rolling, wire drawing, or swaging.

8. Superficial hardening can be effected by shot-blast peening or burnishing. These two methods, however, re-

sult in surface roughness which makes their application inconvenient.

9. Other physical properties are affected in the same proportion as hardness, so that in an 18-8 strip rolled at −300 F. and aged at 750 F. the following values can be obtained: tensile strength, 295,000 psi.; yield stress, 290,000 psi.; proportional limit, 264,000 psi.; elongation, 2.0 per cent; reduction of area, 24.8 per cent; and hardness, 607 V.P.N.

10. The response to these treatments can be controlled by adjusting the iron-chromium-nickel ratios as well as by adding as "alloying elements" austenite promoters such as carbon and nitrogen or ferrite promoters such as molybdenum, columbium, and titanium.

11. Some preliminary experiments have shown the possibility of a considerable improvement in the wear resistance of austenitic chromium-nickel steels. However, more refined techniques are necessary to make this effect reproducible.

12. Some exploratory tests indicate that the salt-water corrosion resistance of samples thus treated is not greatly reduced by the special hardening processes described.

13. The subject matter of this paper is covered by a pending patent application.

Acknowledgment:

The authors wish to express their indebtedness to Crane Co. and Westinghouse Electric Corp. for permission to publish the results of their investigations and gratefully to acknowledge the collaboration of several of their colleagues. W. L. Mienhart and J. R. Goldsmith of Crane Co. made hardness tests and photomicrographic studies while A. Lahr and Harold Peterson conducted the friction tests. G. Comenetz, J. C. R. Kelly, and Kenyon McCoy of Westinghouse contributed by carrying out various phases of the experimental rolling and heat treatment.

DISCUSSION

Messrs. Carl H. Samans[1] and F. J. Shortsleeve[1] (*presented in written form*). —The authors have produced some very interesting properties in austenitic chromium-nickel stainless steels by sub-zero working followed by elevated temperature-aging treatments. Through their kindness we have been able to make X-ray diffraction studies of some of their 18-8 alloys after various stages of this hardening process. The zerolled sample had a very faint austenite pattern with the predominate structure being ferritic, undoubtedly formed by a martensitic (shear-type) reaction as the broadness of the lines indicated appreciable distortion. After aging at 750 F. for 4 hr., the austenite lines had disappeared and the ferrite lines had sharpened a little, thus increasing somewhat their apparent intensity; they still were noticeably broad, however. No evidence of carbide lines was found after etching the surface in an effort to concentrate any carbides which might be present. When three specimens were dissolved simultaneously in acid, no residue was secured, so it is doubtful if many carbides actually had been precipitated by the aging treatment.

There is one other explanation for the added hardening during aging which might be considered by the authors, particularly as it probably could occur simultaneously with and in addition to carbide or nitride precipitation. The martensitic transformation incident to the zerolling undoubtedly produces a severely strained "ferrite" lattice, so the ferrite might tend to recrystallize if the temperature were high enough. If so, the apparent precipitate shown in Fig. 2 might be the result of an initial stage of recrystallization, and the 750 F. aging treatment might correspond to the "recovery" or "stress-relief" stage of recrystallization. An appreciable increase in yield strength and hardness would be anticipated from these effects.

This possible explanation might be tested by examining alloys aged at a higher temperature than 750 F. or for longer times than 4 hr. If the explanation is valid, the "softening" that occurs at higher aging temperatures should be accompanied by recrystallization of the ferrite. However, since carbide precipitation also should occur in this temperature range it might not be possible to eliminate this factor from consideration unless the rates of the two reactions differed appreciably.

It should be pointed out also that, for some alloys systems at least, the amount of work-hardening possible in the immediate vicinity of a phase boundary may be abnormally high, exactly as the authors' have shown in Fig. 6 for varying nickel contents and in Fig. 7 for varying chromium contents. The peaks in both figures correspond approximately to phase boundaries, 8 to 9 per cent nickel dividing the γ and $\alpha + \gamma$ fields, and 17 to 18 per cent chromium, the sigma and non-sigma-forming alloys.

Mr. William Binder.[2]—I should like to mention some work which has been carried out at the Union Carbide and Carbon Research Laboratories, Inc., to determine the influence of low-temperature straining on the impact properties of austenitic 18-8 steel.[3] This work was prompted by the discovery that an 18-8 valve stem, after 15 yr. of service in equipment making liquid oxygen, had transformed partially to martensite under the influence of service strains. A detailed investigation showed that strains of the magnitude encountered in service can have only a minor effect on the impact properties of this type of steel.

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[1] Standard Oil Company (Indiana), Whiting, Ind.

[2] Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls, N. Y.

[3] W. O. Binder, "Effect of Cold-Work at Low Temperature on Austenitic 18-8 Steel," *Metal Progress*, August, 1950, p. 201.

To gain some insight as to the microstructure of the steel after transformation, metallographic and X-ray diffraction studies were made. The X-ray data indicated that the structure of the steel was unaffected when strained about 20 per cent at room temperature by tensile loading; however, a small amount of transformation was evident in the microstructure. On the other hand, straining a total of about 20 per cent at −110 F. produced about 30 per cent ferrite, and at −320 F. about 45 per cent ferrite.

Of considerable theoretical interest is that the specimens strained 10 and 20 per cent at sub-zero temperature exhibited a hexagonal phase having the same interatomic spacing as the low-temperature form of cobalt. It was estimated that about 10 per cent of the austenite had transformed in this manner.

The formation of the hexagonal phase seems to be affected by the method of cold working. Only traces of the phase have been observed in samples of 17-7 and 18-8 steel reduced 18 per cent in thickness by cold rolling at −110 F., and it has not been observed in 18-8 type steel hardenable by heat treatment.

The influence of this phase on the properties of the steel is unknown but will require consideration in any theorizing on this subject.

MESSRS. N. A. ZIEGLER AND P. H. BRACE (*authors' closure*).—The only thing we might mention in addition to the subject matter in the paper is the scattering of the points in Figs. 6 and 7, indicating the increase in hardness due (*a*) to sub-zero working and (*b*) to sub-zero working and subsequent aging. This scattering is caused by different amounts of plastic deformation involved. The above figures include the results obtained by rolling and wire drawing as well as those observed in connection with the Charpy impact test pieces. Obviously in the latter case the amount of plastic deformation can not be very well evaluated.

On the other hand, it is quite certain that the shaded area in Fig. 5, indicating the compositional range of "responsive" alloys, is basically correct.

Our test data include some molybdenum- and columbium-bearing compositions. These two elements do not interfere with this phenomenon and in some cases might even be beneficial. The magnitude of hardening due to sub-zero plastic deformation is determined by the relative metastability of austenite, and the latter in turn depends on the chemical composition and the relative balance of austenite *versus* ferrite promoting elements. We have found that about 70 per cent reduction of the original cross-sectional area, performed at temperatures not higher than −100 F. (−73 C.), develops the hardening phenomenon quite effectively. Some further gain follows decreasing cold-working temperatures and further work will be required to learn what is the most desirable range of cold-working temperature from the economic viewpoint.

As of the moment, we still do not know what is the exact nature of the mechanism of this phenomenon and of the resultant products.

From our limited investigations, it would appear that no serious damage to the corrosion resistance results from the processing we have developed. However, we recognize that this is a matter that needs further and careful attention.

In conclusion we would like to thank Messers. Samans, Shortsleeve, and Binder for very interesting remarks regarding the subject matter of our paper.

HARDENED ALLOY STEEL FOR SERVICE UP TO 700 F.*

By G. V. Smith,[1] W. B. Seens,[1] and E. J. Dulis[1]

Synopsis

This paper describes the properties of two steels, S.A.E. 4340 and 0.40 Carbon Ni-Cr-Mo-V, quenched and tempered to a hardness of Rockwell C 43, which were tested in tension at 75, 500, 600, and 700 F., in compression at 75 F., for creep-rupture strength at 500, 600, and 700 F., and for Charpy impact strength within the temperature range 75 F. to −315 F.

In the last decade the properties of metals for service at temperatures in the neighborhood of 400 to 600 F. have become of increasing importance. Temperatures of this order are anticipated in an increasing number of applications such as in parts of aircraft designed to operate at very high speed, and in guided missiles. In such applications, the aluminum and magnesium alloys, conventionally employed, rapidly lose strength as the temperature is increased, and attention has therefore turned to other alloys.

There are several means of obtaining high strength in ferrous alloys for service at moderate temperatures, among which are:

1. *Cold Rolling.*—In this method chemical composition is generally a minor factor since the strengthening conferred by deformation is not markedly affected by alloying. The austenitic steels, however, do strain-harden more rapidly than the ferritic steels, owing to the transformation of austenite to ferrite (martensite) during deformation. Cold rolling has the significant disadvantage that it develops directionality of properties.

2. *Precipitation Hardening,* as in the recently developed stainless W type of alloy.[2]

3. *Martensitic Hardening.*—In this paper the third method of strengthening has been considered. At the outset it became apparent that very little was known as to the particular properties required for proper design. It has been known for perhaps 25 yr. that at relatively high temperatures, creep properties must be considered, whereas at room temperature, the design stress is generally based on the results of short-time tension tests. The range of temperature around 500 F. has not been fully explored for steels, so far as we are aware. However, it has generally been inferred from A.S.M.E. Boiler Code[3] practice, in which permissible working stresses for steels are based on creep data only for service temperatures of 700 F. or greater, that creep is not a design problem when using steel at 500 F. Inasmuch as we could find no data on the creep properties of hardened steel in this temperature range, creep-to-rupture tests, as well as

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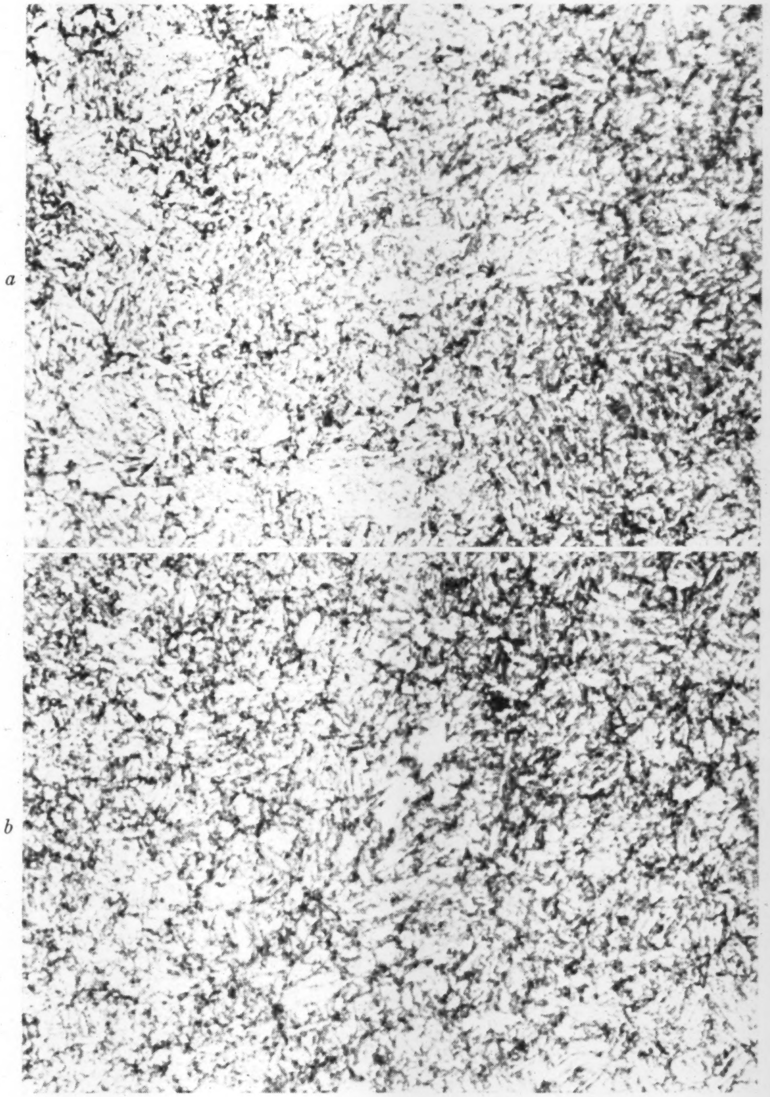
* Presented at the Fifiy-third Annual Meeting of the Society, June 26–30, 1950.

[1] Research Laboratory, United States Steel Corp. of Delaware, Kearny. N J.

[2] R. Smith, E. H. Wyche, and W. W. Gorr, "A Precipitation-hardening Stainless Steel of the 18 per cent Chromium, 8 per cent Nickel Type," *Transactions,* Am. Inst. Mining and Metallurgical Engrs., Vol. 167, pp. 313–345 (1946).

[3] Boiler Code Committee, Am. Soc. Mechanical Engrs., 29 West 39th St., New York, N. Y.

the usual short-time hot tension tests were made. On the basis of initial tests at 500 F., further tests at 600 and 700 F. were made.



(*a*) S.A.E. 4340 Steel Quenched to Martensite after 1 hr. at 1550 F., and Tempered 2 hr. at 850 F.
(*b*) Ni-Cr-Mo-V Steel Quenched to Martensite after 1 hr. at 1600 F., and Tempered 2 hr. at 1050 F.

Fig. 1.—Initial Microstructure of Steels. Etched Picral (×1000).

Compression tests were made at room temperature, but not at the elevated temperatures owing primarily to experimental difficulties. Charpy V-notch impact tests of the quenched-and-tempered material were made at temperatures down to −315 F. to determine relative notch toughness under various conditions, including the effect of prolonged prior exposure at 500 F.

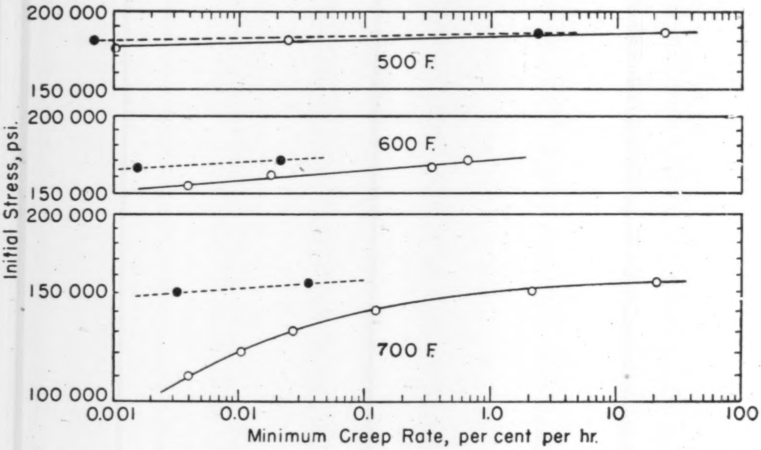


FIG. 2.—Variation of Minimum Creep Rate of S.A.E. 4340 (○——○) and Ni-Cr-Mo-V (●- - -●) Steels with Initially Applied Stress at 500, 600, and 700 F.

Steels were initially quenched and tempered to a hardness of Rockwell C 43 at room temperature.

MATERIALS AND PROCEDURE

The S.A.E. 4340 steel was received in the form of hot-rolled bars, $\frac{1}{2}$ in. square, from a commercial electric furnace heat reported to have the following check analysis:

Heat	No. X21145
Carbon, per cent	0.40
Manganese, per cent	0.70
Phosphorus, per cent	0.009
Sulfur, per cent	0.011
Silicon, per cent	0.29
Nickel, per cent	1.82
Chromium, per cent	0.82
Molybdenum, per cent	0.24

Specimens were machined oversize, heated in cast iron chips (to minimize surface oxidation and decarburization) to 1550 F. for 1 hr. at temperature, which developed an austenite grain size of A.S.T.M. No. 8 to 9, then oil-quenched to 100 per cent martensite. They were tempered in salt at 850 F. for 2 hr. to room temperature hardness of Rockwell C 43 and air-cooled.

The Ni-Cr-Mo-V steel was received in the form of strips $\frac{1}{2}$ in. wide from $\frac{1}{2}$-in. plate which had been rolled from an experimental 300-lb. induction furnace heat made at Battelle Memorial Institute and reported to have the following composition:

Heat	No. A2705
Carbon, per cent	0.40
Manganese, per cent	0.21
Phosphorous, per cent	0.015
Sulfur, per cent	0.015
Silicon, per cent	0.16
Nickel, per cent	2.0
Chromium, per cent	0.97
Molybdenum, per cent	0.37
Vanadium, per cent	0.18
Aluminum, per cent	0.03

Oversize specimens of this steel were machined from longitudinal strips near the quarter line of the width and from transverse strips from the center of the width; they were heat treated as follows: packed in cast iron chips, austenitized 1 hr. at 1600 F., which developed an austenite grain size of A.S.T.M. No. 9 to 10, oil-quenched to 100 per cent martensite (exclusive of undissolved carbides) and tempered at 1050 F. in lead for 2 hr. and water-quenched to final hardness of Rockwell C 43.

After tempering, the specimens of both

steels were ground to final size. Tension specimens were 0.252 in. in diameter for testing with 2-in. gage length extensometers. Elongation was measured over only a 1-in. gage length after fracture; this corresponds to the elongation of the standard 2-in. gage (0.505-in. diameter) specimen. Compression specimens were cylinders 0.250 in. in diameter by 1.000 in. long. Creep-rupture specimens were 0.200 in. in diameter (size limited by testing stand capacity) and had 0.8-in. gage lengths. The Charpy V-notch impact specimens were ground from 0.400 to 0.394 in. square, and the notch (previously milled to a 0.06 in. depth) was

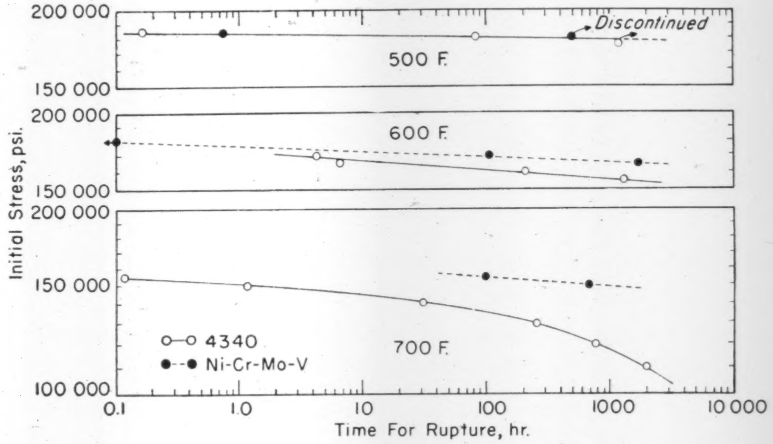


FIG. 3.—Variation of Time to Rupture of S.A.E. 4340 and Ni-Cr-Mo-V Steels with Initially Applied Stress at 500, 600, and 700 F.

Steels were initially quenched and tempered to a hardness of Rockwell C 43 at room temperature.

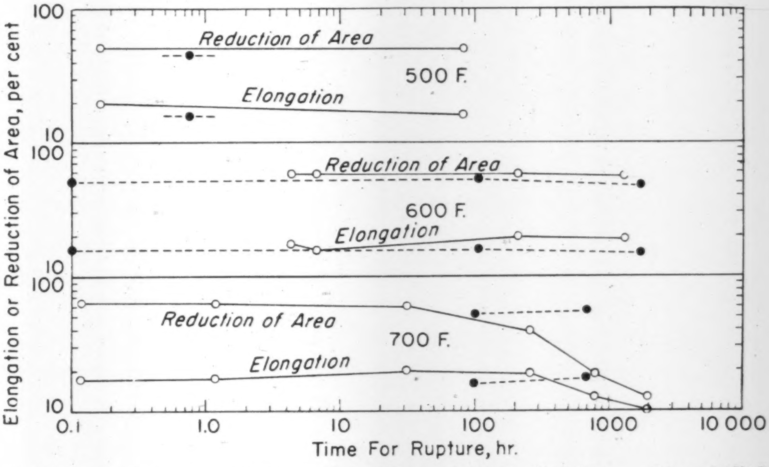


FIG. 4.—Variation of Elongation and Reduction of Area of S.A.E. 4340 (○——○) and Ni-Cr-Mo V (● - - - ●) Steels with Time for Rupture at 500, 600, and 700 F.

Steels initially quenched and tempered to a hardness of Rockwell C 43 at room temperature. Specimens were 0.20 in. in diameter; elongation reported for 0.8 in. gage.

ground to a depth of 0.079 in. All Charpy specimens exposed at 500 F. were finish ground after exposure, except for the specimens of S.A.E. 4340 steel exposed for 500 hr. or less which were finished before exposure.

The tests were made by routine procedure, except in compression for which no procedure had previously been established. Since the small size of the compression specimens prevented direct attachment of an extensometer, a high magnification extensometer was mounted on the columns of the testing machine. The specimen was loaded through hardened steel blocks, so that the indicated strain of the autographic load-deformation diagram included strain in the specimen and elastic strain in the blocks and machine. The specimens showed, however, an abrupt yield point, with deformation under essentially constant load, so that accurate yield strengths could be obtained in spite of the somewhat indeterminate strain scale.

TABLE I.—SUMMARY OF CREEP TO RUPTURE TEST RESULTS.

Steel	Stress to Cause Minimum Creep Rate of 0.01 per cent per hr., psi.			Stress to Cause Rupture in 1000 hr., psi.		
	500 F.	600 F.	700 F.	500 F.	600 F.	700 F.
S.A.E. 4340	178 000	157 000	119 000	179 000	156 000	117 000
Ni-Cr-Mo-V[a]	181 000	168 000	152 000	179 000	166 000	149 000

[a] Longitudinal direction of plate. Transverse direction not tested.

RESULTS AND DISCUSSION

The microstructures of the two steels as quenched and tempered to hardness of Rockwell C 43 (200,000 psi. tensile strength) are quite similar (Fig. 1) and characteristic of tempered martensite. At very high magnification undissolved carbides could be detected in the Ni-Cr-Mo-V steel, especially prior to tempering. These are characteristic of vanadium-containing steels when austenitized at relatively low temperatures, and are presumed to be vanadium carbide (VC).

Creep-to-Rupture Tests:

The results of the creep to rupture tests are given in Figs. 2, 3, and 4 and are summarized in Table I in terms of specific strength criteria commonly employed. Figure 2 shows the variation on log-log coordinates of the minimum creep rate[4, 5] with the initially applied stress. With increasing temperature, the decrease in strength with decrease of creep rate becomes more rapid in either steel, and at the same time, the superiority of Ni-Cr-Mo-V is more evident. At 700 F. several specimens of Ni-Cr-Mo-V failed in the threads, apparently owing to notch sensitivity, and only two within the gage section. Owing to the loss of specimens in tests at 700 F. there were only two remaining for test at 600 F.

The curvilinear relation for log-log plotting observed at 700 F. for the variation of creep rate, as also for time to rupture (Fig. 3), with initial stress in the case of the S.A.E. 4340 steel (and possibly also for the Ni-Cr-Mo-V steel if more data were available) is of interest, for it contrasts with the essentially linear variation observed at lower temperature for this material and in numerous other tests in this laboratory for a wide variety of steels at different temperatures. Semi-log plotting does not result in linear variation at 700 F.

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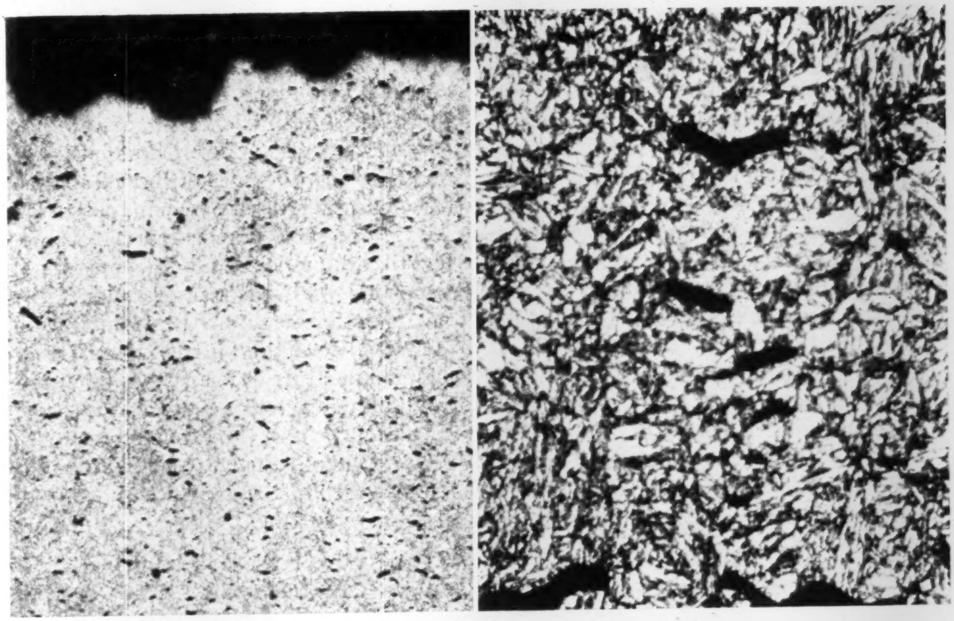
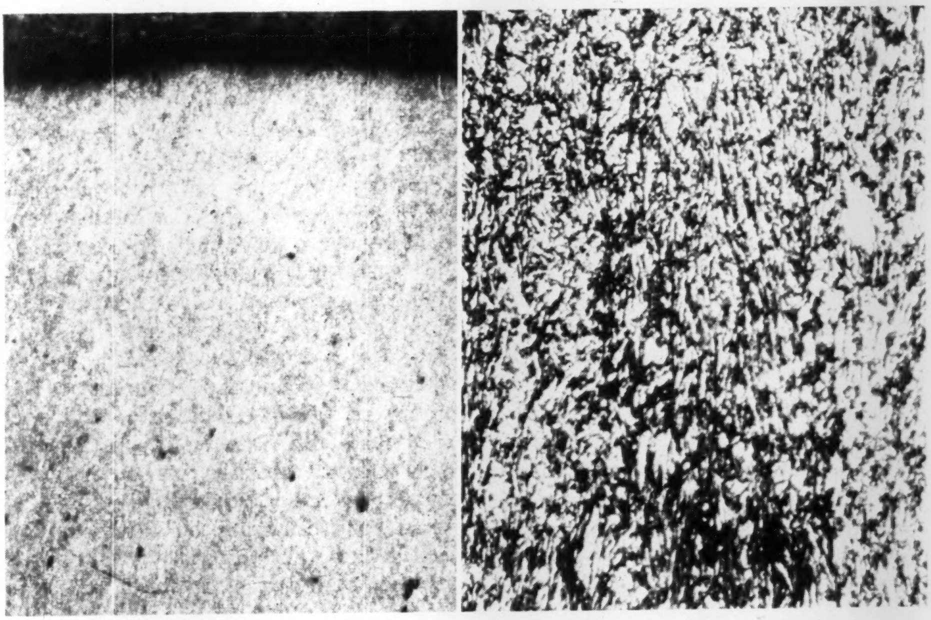
[4] The rate of creep in a constant load creep-to-rupture test in tension characteristically decelerates during the initial stage, then remains substantially constant and minimum, and finally accelerates with impending fracture. Creep deformation is quite commonly evaluated in terms of the stress to produce a specific minimum-stage creep rate, although such practice ignores the creep occurring during the initial stage.

[5] G. V. Smith, E. J. Dulis, and E. G. Houston, "Creep and Rupture of Several Chromium-Nickel Austentitic Stainless Steels," *Transactions*, Am. Soc. Metals, Vol. 42, pp. 935–980 (1950).



(*a*) 1920 hr. to rupture under 110,000 psi.



(*b*) 0.1 hr. to rupture under 155,000 psi.

Fig. 5.—Microstructural Characteristics of Fracture in Long- and Short-Time Creep to Rupture Tests of S.A.E. 4340 Steel at 700 F. Picral Etch. Longitudinal Sections at or Near Fracture.

The slope of the plot of time for rupture *versus* initial stress increases with increasing temperature (Fig. 3), and the superiority of the Ni-Cr-Mo-V becomes greater. Although at 500 F. both steels required essentially the same stress, 179,000 psi., for rupture in 1000 hr., at 700 F. the Ni-Cr-Mo-V required a stress for rupture in this time some 25 per cent greater than the S.A.E. 4340 steel, 149,000 and 117,000 psi., respectively. At the intermediate temperature of 600 F. the difference was less pronounced, 166,000 against 156,000 psi.

Figure 4 shows the variation of elongation and reduction of area with time to rupture. Except in the case of the S.A.E. 4340 steel tested at 700 F., no marked change in ductility was encountered within the test limits. In this exception the elongation and reduction of area fell off significantly beyond some 500 hr.[5, 6] This decrease was associated with a change in the character of the fracture as revealed in the photomicrographs of Fig. 5. These show longitudinal sections at or near the fracture, at low and high magnification, for the S.A.E. 4340 steel after fracture in the longest and shortest time intervals at 700 F., 1920 and 0.1 hr., respectively. Many transverse cracks and little distortion of the grain structure are apparent in the long-time fracture; in the short-time fracture these cracks are not evident and the grain structure is distorted in the direction of tension. These features may be seen by comparison with Fig. 1. The short-time fracture is characteristic of ductile metals at atmospheric temperature, that is transgranular, while the long-time fracture is characteristic of creep deformation involving relatively high temperature or relatively slow strain rate, that is intergranular. Either type may occur at the same temperature dependent upon the rate of strain. No microstructural change, such as spheroidization, was noted.

TABLE II.—RESULTS OF SHORT-TIME TENSION TESTS.
(Average of 2 tests 0.25 in. in diameter)

Temperature, deg. Fahr.	Steel	Yield Strength, 0.2 per cent offset, psi.	Tensile Strength, psi.	Elongation in 1 in., per cent	Reduction of Area, per cent
75	S.A.E. 4340	187 700	198 100	14.0	53.0
75	Ni-Cr-Mo-V (Longitudinal)	189 900	199 600	17.0	53.5
75	Ni-Cr-Mo-V (Transverse)	189 200	198 800	15.0	47.5
500	S.A.E. 4340	155 700	190 800	20.5	59.0
500	Ni-Cr-Mo-V (Longitudinal)	156 000	184 100	17.0	55.0
500	Ni-Cr-Mo-V (Transverse)	157 000	185 400	16.0	49.0
600	S.A.E. 4340	146 600	177 200	21.0	64.5
600	Ni-Cr-Mo-V (Longitudinal)	148 800	176 700	18.0	58.0
700	S.A.E. 4340	138 000	163 800	20.0	71.0
700	Ni-Cr-Mo-V (Longitudinal)	145 000	169 400	17.5	59.0

In connection with the creep-to-rupture results, it should be pointed out that in general it is highly desirable to determine the relation between stress and total creep rather than simply the minimum creep rate as shown in Fig. 2. This can be conveniently accomplished in terms of the intercept deformation as described in an earlier publication.[5] In the present investigation, which was undertaken as an exploratory survey, the extensions occurring on loading could not be determined precisely owing to the method of measuring extension by means of the displacement of the loading lever,[7] and to the very large deformations involved, and are therefore not reported.

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[6] A plot of stress *versus* the ratio of elongation at fracture to time at fracture, which in all other instances examined has been found to be linear on log-log coordinates and thus suitable for predicting elongation at rupture, was found for these data to be curved in the same manner a was the minimum creep rate plot of Fig. 2.

[7] G. V. Smith, W. G. Benz, and R. F. Miller, "Creep and Creep-Rupture Testing," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, pp. 615–638 (1947).

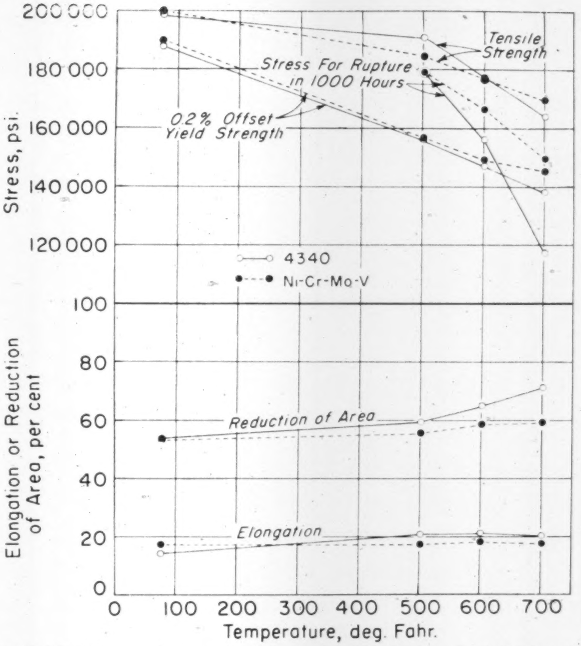

FIG. 6.—Variation of Yield Strength, Tensile Strength, Elongation, and Reduction of Area of S.A.E 4340 and Ni-Cr-Mo-V Steels Determined in Short Time Tension Tests at 75, 500, 600, and 700 F.

Also shown is the stress to cause rupture in 1000 hr. as taken from Fig. 3. Steels initially quenched and tempered to a hardness of Rockwell C 43 at room temperature.

TABLE III.—RESULTS OF COMPRESSION TESTS AT 75 F.
(Average of 4 specimens)

Steel	Yield Strength, 0.2 per cent offset, psi.
S.A.E.	196 500
Ni-Cr-Mo-V (Longitudinal)	202 200
Ni-Cr-Mo-V (Transverse)	201 000

TABLE IV.—RESULTS OF CHARPY V-NOTCH IMPACT TESTS.
(Observed Values in ft-lb.)

Test Temperature, deg. Fahr.	S.A.E. 4340	Ni-Cr-Mo-V	
		Longitudinal	Transverse
75	28	22	18
	28.5	22.5	18
		23	18.5
0	24.5	21	16.5
	24.5	22	18
	23.5	22	17
−25	21	20.5	...
	22	20.5	...
	21	20	...
−50	18.5	19	...
	17.5	20	...
	18	19.5	...
−100	16	16.5	14.5
	16	16	(jammed)
	16	16.5	(jammed)
−200	14	14.5	13.5
	13.5	15	12.5
	13	15.5	12.5
−315	11.5	10	...
	10	9	...
	9	10	...

In several instances the elastic and plastic extension occurring on or shortly after application of load amounted to 2 to 3 per cent, as a consequence of the very high stresses which necessarily had to be employed in order to break the samples. At the lower temperatures these stresses were in most cases not far below the tensile strength determined in the usual short-time tension test. Because of this, it appeared that the creep data would not be employed in design, and the accurate

TABLE V.—RESULTS OF CHARPY V-NOTCH IMPACT TESTS AFTER EXPOSURE AT 500 F. IN AIR.
(Observed Values in ft-lb.)

Test Temperature, deg. Fahr.	Exposure Period			
	None	220 hr.	500 hr.	1000 hr.
	S.A.E. 4340			
75 to 85......	28	28	28	27
	28.5	27	27.5	26
	...	28.5	26	27.5
−315.........	11.5	10	10	11
	10	9	10	11
	9	10.5	9	11.5
	Ni-Cr-Mo-V (Longitudinal)			
75 to 85......	22	22.5	26.5	25.5
	22.5	23	23	24.5
	23	25	25	25
−315.........	10	10	9	12
	9	9.5	11.5	13
	10	13	10	(defect)

determination of the initial strains was not undertaken.

Short-Time Tension Tests:

The results of the short-time tension tests (with good agreement of duplicate tests) are presented in Table II and plotted in Fig. 6 (longitudinal results only). Figure 6 also shows the creep-rupture results for comparison; this comparison will be discussed below. With increasing temperature, the yield and tensile strengths of both steels decrease while the elongation and reduction of area either remain unchanged or increase slightly. Although the two steels have essentially the same strength at room temperature, Ni-Cr-Mo-V steel is slightly superior at the maximum test temperature, 700 F.

Transverse as well as longitudinal tests were made at 75 and 500 F. The transverse properties were essentially identical with the longitudinal except in respect to the reduction of area which was about 10 per cent less for the transverse direction.

Compression Tests:

The results of the compression tests at 75 F. are given in Table III. The compressive yield strength of the S.A.E. 4340 steel exceeded the tensile yield strength by about 5 per cent and approximated the tensile strength. For the Ni-Cr-Mo-V steel, the compressive yield strength slightly exceeded the tensile strength. As in the case of the tension tests, the com-

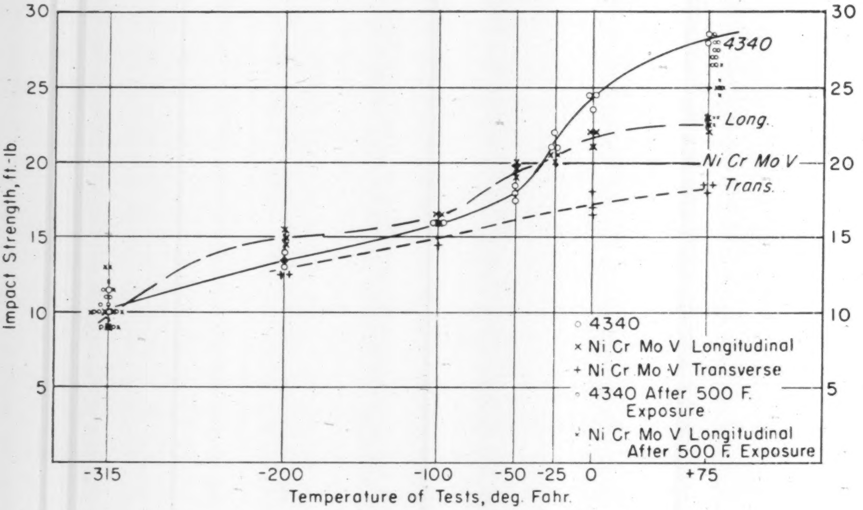

FIG. 7.—Charpy V-Notch Impact Tests of S.A.E. 4340 and Ni-Cr-Mo-V Steels.

pressive offset yield strength corresponds to the lower yield point, although in some of the tests, the slight drop in load beyond the yield point was too small to show on the recorder diagram. The difference between longitudinal and transverse directions was only 1 part in 200. Excellent reproducibility was obtained in the tests of the four specimens of each group with maximum variation of only 500 psi.

Notch Impact Tests:

The results of the notch-impact tests are tabulated in Tables IV and V and are plotted in Fig. 7. There was little difference between the two steels at the lower temperatures, but at room temperature the S.A.E. 4340 steel was superior to the Ni-Cr-Mo-V steel. At −200 F. the transverse values for Ni-Cr-Mo-V steel were only slightly lower than longitudinal values, whereas at 0 F. and 75 F. the directional effect was much greater. In all the Ni-Cr-Mo-V specimens the axis of the notch was perpendicular to the plate surface to reduce possible effects of laminations; however, no such defects were encountered except in one specimen, tested at room temperature after exposure to 500 F. for 1000 hr. As that specimen appeared to have a small crack in the fracture, the test was rejected.

The change of slope of the curve for S.A.E. 4340 steel between −50 F. and 0 F. is not believed to indicate a transition temperature range, because there was no corresponding change in the appearance of the fractured specimens. All fractures of both steels had the dull, fine grain, smooth texture typical of tempered martensite. The side "cup" (so-called due to similarity to the cup and cone appearance in tensile fractures) decreased in regular manner with lower test temperatures. At room temperature the fractures were about 65 per cent plane, whereas at −315 F. the fractures were over 95 per cent plane.

The smaller symbols in Fig. 7 indicate the results of Charpy tests after exposure to 500 F. but without differentiation of the different duration of exposure which may be studied with less confusion from Table V. For the S.A.E. 4340 steel there was no significant effect of exposure other than a lowering of the over-all group average by about 1 ft-lb. at room temperature. For the Ni-Cr-Mo-V steel, Table V indicates a slight upward trend in values with increasing duration of exposure for tests at both room temperature and −315 F.

The hardness of the specimens was determined after exposure at 500 F. and was identical with that before exposure.

GENERAL DISCUSSION

The stresses for rupture in 1000 hr. for both steels, taken from Fig. 3, have been plotted on Fig. 6 for ready comparison with the short-time yield and tensile strengths. At 500 F. this rupture strength is considerably greater than the yield strength and not far below the tensile strength. Accordingly, it would appear that no cognizance need be taken of creep in the design of structures to operate at this temperature, at least for periods not exceeding 1000 hr., and, most probably, even for very long intervals, since the plot of stress *versus* rupture time is quite flat and the creep-rates quite small or undetectable. This conclusion is based on the assumption that stresses in excess of the yield strength would not be employed.

At 600 F. the stress for rupture in 1000 hr. still exceeds the yield strength for both steels, but at 700 F. this stress is some 20,000 psi. less than the yield strength for the S.A.E. 4340 steel, though still slightly greater for the Ni-Cr-Mo-V steel. Thus as the temperature is increased to 700 F. the phenomenon of creep becomes increasingly important and at this latter temperature should be considered for certain applications, at least for the S.A.E. 4340 steel.

The actual basis of design in any specific case must depend upon the application and take into account such factors as the expected life and tolerable deformation.

SUMMARY

With increasing temperature, the yield and tensile strengths of both steels decreased (Fig. 6). In the case of the Ni-Cr-Mo-V steel which was received in the form of plate, both longitudinal and transverse tests were made at 75 and 500 F. but little difference in properties was detected. Although the two steels were of approximately equal yield or tensile strength at room temperature, the Ni-Cr-Mo-V steel was slightly stronger at the highest test temperature, 700 F. With increasing temperature, the elongation and reduction of area in short-time tension tests remained substantially unchanged or increased slightly.

In the creep-to-rupture tests, the strength, measured either in terms of the stress to cause rupture in a specific time or of the stress to produce a specific minimum creep rate, also decreased with increasing temperature. With increasing temperature, the creep-rupture strength, as expressed, for example, as the stress to cause rupture in 1000 hr., Fig. 6, decreased more rapidly than the yield and tensile strengths, and the superiority of the Ni-Cr-Mo-V steel at the two highest test temperatures was more marked than in the short-time tests. In the case of the S.A.E. 4340 steel tested at 700 F. this stress had fallen 20,000 psi. below the yield strength at this temperature, whereas for the Ni-Cr-Mo-V steel, it was still slightly greater. The elongation and reduction of area fell off sharply for the S.A.E. 4340 steel in the longer creep-to-rupture tests at 700 F. (Fig. 4) and the type of fracture changed from transgranular to intergranular. Many of the Ni-Cr-Mo-V steel samples tested at this temperature failed at the root of a thread, indicating notch sensitivity, and only two specimens failed in the gage section; the reduction of area and elongation of the sample requiring the longer time to rupture, however, did not show any tendency to decrease, as with the S.A.E. 4340 steel.

Compression tests of the two steels at room temperature showed the yield strength of either steel to be slightly greater (about 5 per cent) than the tensile yield strength, and in the case of the Ni-Cr-Mo-V steel slightly greater than the ultimate tensile strength. Tests in the transverse direction of this steel showed the difference from the longitudinal yield strength to be negligible.

Charpy V-notch impact tests from room temperature down to −315 F. showed gradual loss in toughness with lowered temperature (Fig. 7). The two steels had similar notch impact strength below 0 F, but above this temperature the S.A.E. 4340 steel was superior. Tests at 75 and −315 F. after exposure at 500 F. for periods up to 1000 hr. showed no effect from such exposure for S.A.E. 4340 steel and a slight but irregular increase in values for Ni-Cr-Mo-V steel. The minimum Charpy value (V-notch specimen) was 9 ft-lb. at −315 F.

Tests of both longitudinal and transverse specimens of Ni-Cr-Mo-V steel showed the notch impact strength to be lower for transverse than for longitudinal specimens.

When it is desired that the design stress not exceed the short-time tensile yield strength, it would appear that no cognizance of creep need be taken for these materials for temperature below about 700 F., at least for service not exceeding 1000 hr., and most probably for much longer intervals.

Acknowledgments:

The steels were obtained for us by P. R. Wray and J. M. Hodge of Carnegie-Illinois Steel Corp., who also gave helpful advice concerning the test program. We wish also to acknowledge the assistance of E. G. Houston and W. L. Jensen.

DISCUSSION

Mr. J. W. Freeman.[1]—I should like to ask Mr. Smith one question: Does he have any opinion as to how much effect the long-time exposure to temperature during the test had on the loss of strength? That is, was the rather surprising amount of creep at these low temperatures due to loss of strength by tempering rather than by creep in the ordinary way?

Mr. C. T. Evans, Jr.[2] (*by letter*).—This is an interesting piece of exploratory work in a field where there has been much conjecture and few facts. Information on behavior of highly stressed materials in the temperature range of room temperature to 1000 F. is always welcome to the steam turbine industry and is also applicable to gas turbine design. For instance, in some designs a gas turbine disk with a rim temperature of around 1100 F. will only be at about 600 F. at the hub where the stresses are at a maximum. The idea of using so-called "ferritic" materials (such as S.A.E. 4340) for these disks is quite attractive from production and strategic viewpoints.

As we see it, neither the S.A.E. 4340 nor Ni-Cr-Mo-V materials come through the authors' tests with a clean bill of health. The S.A.E. 4340 shows a somewhat alarming decline in rupture strength and ductility at 700 F., with attendant microstructural cracking. On the other hand, the Ni-Cr-Mo-V material held up well in strength at 700 F. for the two specimens which fractured in the gage length, but most of the specimens were lost by cracking through the threads. The authors indicate that this may be due to notch-sensitive behavior. Since practically all engineering structures have "notches" (for example, the pine tree blade fastening of a turbine disk) this phenomenon is quite alarming.

We have been doing some work on stress rupture of notched specimens of superalloys, using the specimen shown in Fig. 8. This specimen produces a known degree of triaxiality in the test—approximately 1.47 as defined by Sachs, Lubahn, and Ebert[3] as the ratio of the rupture strength with a notch (based on nominal area beneath the notch) to the rupture strength without notch. It is suggested that comparison rupture tests with a specimen of this type would be a better criterion of notch sensitivity than the observation that standard specimens failed in the threads, since undoubtedly the threads were not made to any known radius.

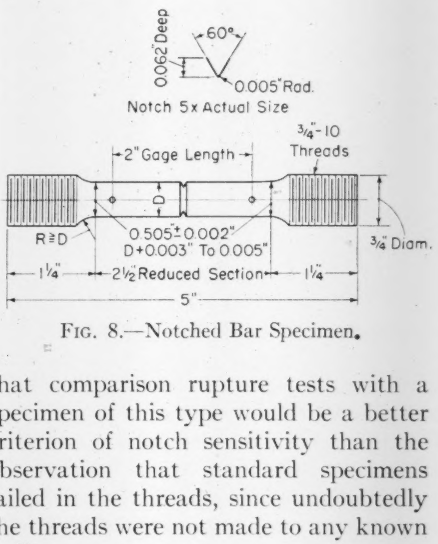

Fig. 8.—Notched Bar Specimen.

The authors do not suggest any explanation for the microstructural cracking which is apparently the cause of the decline in rupture strength and ductility of the S.A.E. 4340 at 700 F. It has been suggested that this may be due to oxidation effects. If so, the point could be quite readily established by running some tests under nitrogen atmosphere.

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[2] Chief Metallurgist, Elliott Co., Jeannette, Pa.

[3] G. Sachs, J. D. Lubahn, and L. J. Ebert, "Notched Bar Tensile Test Characteristics of Heat Treated Low Alloy Steels," *Transactions*, Am. Soc. Metals, Vol. 33, p. 340 (1944).

MESSRS. G. V. SMITH, W. B. SEENS, AND E. J. DULIS (*authors' closure*).—In exploration of the question raised by Mr. Freeman as to whether the test materials softened during test as a result of additional tempering, we have measured the hardness in the plastically undeformed shoulder section of the specimen of each steel tested for the longest time at 700 F. The Ni-Cr-Mo-V steel which broke in 680 hr. at this temperature had a shoulder hardness of 441 DPH (diamond pyramid hardness) compared with an initial hardness of 428 DPH; the 4340 steel after rupture in 1920 hr. had a shoulder hardness of 420 DPH compared with an initial value of 428 DPH. In either case, the change is probably not significant. Since these steels undergo secondary hardening, and thus may exhibit a relatively complex tempering behavior, a complete answer to Mr. Freeman would require the study of the effect of time as well as of the influence, if any, of stress and plastic deformation on the tempering reaction.

Mr. Evans' comments do not appear to require reply. As he recognizes, our work was of an exploratory nature, and much additional testing of these steels as well as others, especially in other conditions of heat treatment, is to be desired.

EFFECT OF TEMPERATURE ON THE MECHANICAL PROPERTIES, CHARACTERISTICS, AND PROCESSING OF AUSTENITIC STAINLESS STEELS*

By V. N. Krivobok[1] and A. M. Talbot[2]

Synopsis

In this paper are presented the results of investigations dealing with the processing (such as rolling) of commercial austenitic stainless steels at sub-zero temperatures showing that such processing enhances the mechanical properties and improves other characteristics of the steels. The latter were represented by the most popular grades such as A.I.S.I. 301, 302, 304, 310, and 347.

The mechanical properties at sub-zero and at elevated temperatures were determined for steels rolled according to normal practice (at room temperature) and for steels rolled at sub-zero temperatures. The effects of sub-zero and of elevated temperatures on the properties at room temperature were also determined.

The uncovered improvement in ductility characteristics, under conditions of sub-zero temperatures, of steels strengthened by rolling is discussed in terms of its relation to the forming process.

An early study of the influence of sub-zero-temperatures on the properties of certain steels was that of Colbeck, MacGillivray and Manning (**1**).[3] These steels included chromium-nickel steels in the quench-annealed condition. They observed that as *low temperatures were approached* (from room temperature down to −290 F.) the tensile strength was prominently increased, the yield point improved (although to a lesser degree than tensile strength), and ductility generally lowered. These observations have been confirmed by more recent investigations. The lowering in ductility, that is, elongation in 2 in. and reduction of area, was influenced by the composition of each stainless steel tested. In fact, in the case of one steel, namely, that with 12.25 per cent chromium and 16 per cent nickel, both elongation and reduction of area at −290 F. were somewhat *higher* than at room temperature. The impact properties of the steels remained on the high level, showing no presence of brittleness that is characteristic of carbon and low-alloy steels.

The Monograph on "Alloys of Iron and Chromium" (**2**) reports the work of de Haas and Hatfield, also of Russell and co-workers: both investigations uncovered the same phenomenon—a substantial increase in tensile and yield strengths as the testing temperatures were lowered respectively, to −355 F. and −40 F. In the first instance the increase in tensile strength was more than 250 per cent while that in yield strength was only 100 per cent.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 928.

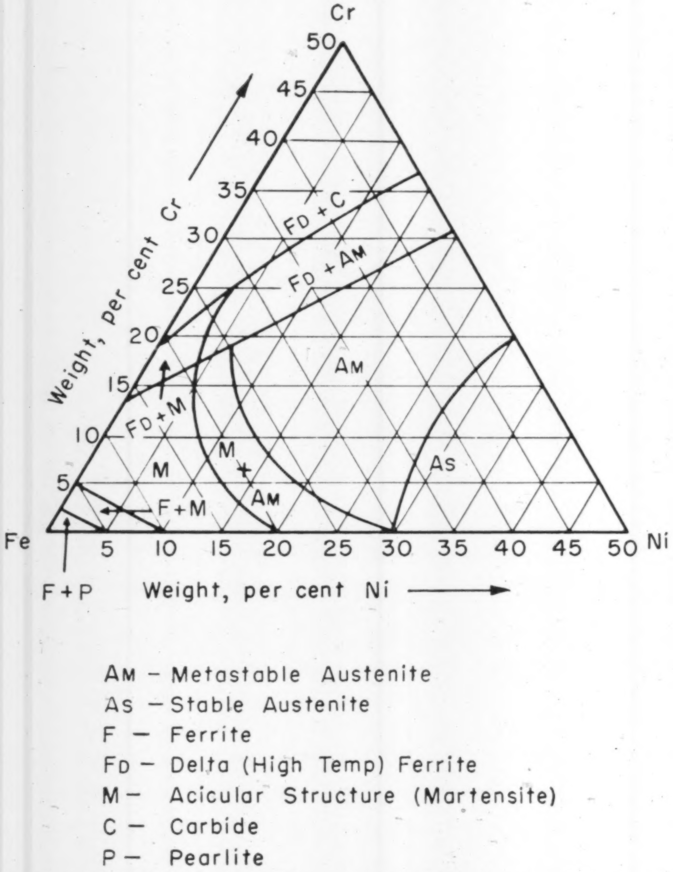
Cr
50
45
40
35
30
25
20
15
10
5
Fe
5
10
15
20
25
30
35
40
45
50
Ni
Weight, per cent Cr
Weight, per cent Ni
F + P
FD + C
FD + AM
AM
FD + M
M
M + AM
F + M
As
AM – Metastable Austenite
As – Stable Austenite
F – Ferrite
FD – Delta (High Temp) Ferrite
M – Acicular Structure (Martensite)
C – Carbide
P – Pearlite

FIG. 1.—Phases After Rapid Cooling from Temperatures of Maximum γ (0.1 per cent C) (*Metals Handbook*, 1948 Edition, p. 1261).

S. J. Rosenberg (3) also studied the effect of low temperatures on the properties of several ferrous and non-ferrous alloys. Quite recently a comprehensive study of the various mechanical properties of stainless steels at sub-zero temperatures was presented by McAdam, Geil and Cromwell (4). Both of these valuable contributions substantiated previous findings—increased strength, both static and dynamic, retention of satisfactory ductility and toughness at temperatures as low as −306 F. (the lowest temperature studied by McAdam and his co-workers).

In all of the above cited work, most of the mechanical testing was carried out on steels which were rendered "soft" and ductile by a previous quench-annealing heat treatment. In a few instances cold-worked steels were included in the investigations (Rosenberg, McAdam, *et al.*)

with substantially the same results as summarized above. It is to be noted that cold working of the steels was accomplished at *room* temperature, and only *testing* was done at temperatures below zero.

The effect of mechanical working at room temperature has been well studied and need not be described here. Comparatively little work has been done on the possible effect of exposure to subzero temperatures. Such studies would obviously include the determination of mechanical properties *before* and *after* the steels in question were subjected to the influence of extremely low temperatures for various lengths of time.

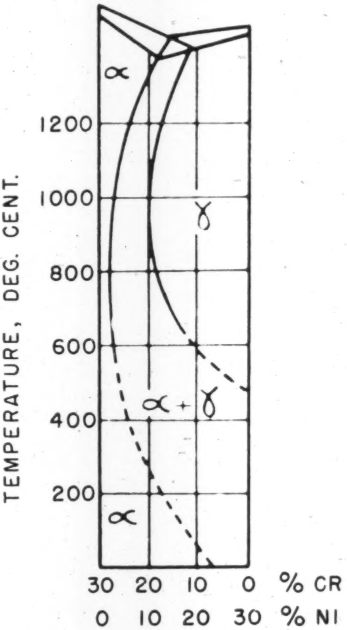

FIG. 2.—Cross-Section of Fe-Cr-Ni System at 70 per cent Fe. From "A Study of the Iron-Chromium Nickel System" by J. W. Pugh and J. D. Nisbet, *Journal of Metals*, Vol. 188, No. 2, p. 272 (1950).

Chromium-nickel stainless steels of standard compositions are, essentially, alloys of iron, chromium and nickel. While it is true that carbon, even in small amounts, has a marked influence on the structural constituents that can be developed in chromium-nickel stainless steels by appropriate heat treatments, the main characteristics of these steels are adequately presented by the ternary system Fe-Ni-Cr (5) as shown in Figs. 1 and 2 (cross-section of the same system at 70 per cent Fe) (6). The observation of immediate interest to this work is that all of the chromium-nickel stainless steels, rapidly cooled from temperature of "maximum gamma" (austenite, 0.1 per cent C), are represented, at room temperature, by *metastable* austenite. The use of the word "metastable" is deliberately chosen. It denotes the fact that such austenite is not a phase in a physico-chemical sense because it does not maintain its identity and characteristics under all conditions of heat or mechanical treatment. The metastability of austenite (sometimes referred to as "instability") is relative—that is, the chemical composition of austenite affects its tendency to be transformed into ferrite by mechanical means at room temperature, or without mechanical means by merely lowering the temperature. Published data on the latter possibility is not abundant. But even from the scant data, we know that structural and phase changes in the *least stable* austenite may be brought about by exposure to low temperatures.

There is some evidence, for example, that when a commercial alloy of type 301 stainless steel was held at −320 F. for a considerable length of time, it showed a noticeable increase in its magnetic permeability.

Earlier experiments conducted by one of the authors (7) on experimental and commercial alloys of iron, chromium, and nickel showed, conclusively, that *it is possible* to produce, by selection of an appropriate analysis and heat treatment, an austenite of such low degree of stability that it would transform if subjected to low temperatures. In other words, it can be proven that "low stability" austenite can be produced by quenching from elevated temperatures and that such austenite remains unchanged at room temperature: when submersed into liquid air (or even that of carbon dioxide) it undergoes fast decomposition, accompanied by various degrees of hardening.

TABLE I.—EFFECT OF SUB-ZERO TEMPERATURE TREATMENT ON HARDNESS AND MAGNETIC PROPERTIES OF NICKEL BEARING 18 PER CENT CHROMIUM STEELS.

After Krivobok and Gensamer (7).

Alloy	Main Alloy Composition, per cent			Rockwell Hardness						Magnetic[a] Characteristics	
				Heated to 1850 F.				Heated to 2300 F.		Heated to 2300 F.	
	Carbon	Chromium	Nickel	Air Cooled	Air Cooled and Cold Treated (−320 F.)	Water Quenched	Water Quenched and Cold Treated (−320 F.)	Water Quenched	Water Quenched and Cold Treated (−320 F.)	Water Quenched	Water Quenched and Cold Treated (−320 F.)
A	0.05	17.4	2.0	...	...	17C	22C	16C	26C	N.M.	M
B	0.17	17.1	2.2	...	...	...	...	32C	31C	M	M
C	0.27	18.4	2.0	...	...	44C	51C	37C	48C	M	M
D	0.60	18.1	2.0	58C	58C	...	...	21C	44C	S.M.	M
E	0.05	17.4	4.0	...	...	...	...	17C	28C	N.M.	M
F	0.16	17.7	4.2	22C	28C	...	...	14C	35C	N.M.	M
G	0.10	17.6	5.0	35C	43C	...	...	17C	37C	N.M.	M
H	0.05	17.2	7.8	...	...	...	...	80B	88B	N.M.	N.M.
I	0.23	17.8	8.0	...	...	...	...	85B	85B	N.M.	N.M.
J	0.34	17.6	8.1	...	...	...	...	86B	83B	N.M.	N.M.
K	0.05	17.8	11.4	...	...	...	...	78B	78B	N.M.	N.M.
L	0.20	18.0	12.0	...	...	...	...	79B	78B	N.M.	N.M.

[a] M = Magnetic
N.M. = Non-magnetic
S. M. = Slightly magnetic.

This observation is illustrated by alloys E, F, and G to be found in Table I. Upon rapid cooling from high temperatures, these alloys have hardness characteristics of austenite in stainless steels and are practically non-magnetic (permeability between 1.003 and 1.06). Upon being subjected to the temperature of liquid nitrogen, the hardness of these alloys is appreciably increased while a readily detectable increase in magnetism is noted. As the stability of austenite is raised,—and it is effectively done by increasing contents of nickel (alloys H and K) in otherwise essentially the same composition, no hardening effects by low temperatures are observed and no change in magnetic properties takes place.

EFFECT OF HOLDING AT SUB-ZERO TEMPERATURES ON THE PROPERTIES, AT ROOM TEMPERATURE, OF STANDARD AUSTENITIC STAINLESS STEELS

To study the effect of exposure to low temperatures on the properties of standard chromium-nickel stainless steels, several such steels were tested. Room temperature tests of samples exposed to temperatures down to −105 F. furnishes conclusive data that holding as long as 100 hr. at that temperature produced no adverse change in the tensile properties of stainless steels either annealed or cold worked. These data are shown in Table II. This is true even in the case of the least stable type 301 which previously had been mentioned as changing its magnetic properties at −320 F. Table III gives the compositions of these steels.

Other supplementary experiments show that annealed stainless steel types 302, 304, and 310 undergo no change in their room temperature *impact properties* even though they may be subjected for a considerable length of time to extremely low temperatures such as are encountered at −320 F. Data concerned with the impact characteristics of type 304 are reproduced in Table IV.

TABLE II.—EFFECT OF HOLDING AT SUB-ZERO TEMPERATURES ON TENSILE PROPERTIES.

Type	Condition	Treatment Prior to Testing	Tensile Properties		Elongation in 2 in., per cent
			Yield Strength 0.2 per cent offset, psi.	Tensile Strength, psi.	
301	Annealed	None	35 000	120 000	68
301	Annealed	None	35 000	119 000	69
301	Annealed	100 hr. at −105 F.	38 000	129 000	73
301	Cold rolled	None	114 000	156 000	31
301	Cold rolled	None	114 000	155 000	25
301	Cold rolled	100 hr. at −105 F.	115 000	162 000	32
301	Cold rolled	None	184 000	208 000	14
301	Cold rolled	None	175 000	210 000	14
301	Cold rolled	100 hr. at −105 F.	170 000	210 000	15
302	Annealed	None	34 000	89 000	67
			34 000	89 000	62
302	Annealed	100 hr. at −105 F.	34 000	88 000	68
302	Cold rolled	None	150 000	155 000	14
			150 000	156 000	12
302	Cold rolled	100 hr. at −105 F.	152 000	156 000	14
347	Annealed	None	39 000	95 000	49
			39 000	95 000	48
347	Annealed	100 hr. at −105 F.	41 000	99 000	49
347	Cold rolled	None	140 000	153 000	8
			139 000	154 000	8
347	Cold rolled	100 hr. at −105 F.	137 000	152 000	8

NOTE.—All the above tests were carried out on standard 0.5 in. wide samples of 0.023 to 0.030 in. thickness. All tests were performed at room temperature.

TABLE III.—CHEMICAL COMPOSITION OF MATERIALS TESTED.

Material	Stock	Composition, per cent					
		Carbon	Manganese	Silican	Nickel	Chromium	Columbium
Commercial annealed type 301	No. 02281	0.10	1.06	0.42	7.13	17.57	....
Commercial full hard type 301	No. 02281	0.10	1.06	0.42	7.13	17.57	....
Commercial half hard type 301	No. 02280	0.10	1.06	0.50	7.63	17.20	....
Commercial annealed type 302	No. 02289	0.10	0.45	0.37	9.15	19.03	....
Commercial half hard type 302	No. 02289	0.10	0.45	0.37	9.15	19.03	....
Commercial annealed type 347	No. 02285	0.06	1.39	0.44	9.90	17.69	0.59
Commercial half hard type 347	No. 02285	0.06	1.39	0.44	9.90	17.69	0.59
Type 301 used for sub-zero rolling	No. 02553	0.08	0.73	0.54	7.49	17.72	....
Type 301 used for sub-zero rolling	No. 02672	0.111	1.35	0.53	7.45	17.66	....
Type 302 used for sub-zero rolling	No. 02550	0.12	....	....	9.30	18.30	....
Type 302 used for sub-zero rolling	No 02673	0.113	1.12	0.63	9.95	17.88	....
Type 302 used for sub-zero rolling	No. 02716	0.12	0.63	0.37	9.16	18.37	....
Type 304 used for sub-zero rolling	No. 02551	0.071	....	....	9.48	18.10	....
Type 304 used for sub-zero rolling	No. 02670	0.072	1.17	0.74	9.87	18.52	....
Type 347 used for sub-zero rolling	No. 02552	0.07	....	....	11.17	17.74	1.00
Type 347 used for sub-zero rolling	No. 02674	0.058	1.28	0.45	10.64	18.01	0.78
Type 310 used for sub-zero rolling	No. 02549	0.046	....	....	20.87	24.44	....

Since a change in mechanical properties would be as readily detected by impact testing as by tension tests, we may accept the above as additional evidence that mere exposure to low temperatures, within the ranges studied, does not adversely affect the room-temperature properties of commercial austenitic stainless steels, either quench-annealed or cold worked. The composition of these steels, that is the amount of alloying elements, is such as to insure retention of metastable austenite under a range of temperatures, probably even considerably lower than those investigated.

EFFECT OF SUB-ZERO TEMPERATURES AND MECHANICAL WORKING ON THE PROPERTIES OF STAINLESS STEELS

Above we discussed the *separate* effects of either mechanical working or exposure to low temperatures. The purpose of the experimental work, described in detail on the following pages, was to ascertain the combined effects of working *and sub-zero* temperatures (down to −320 F.) on the decomposition of austenite, that is, on its mechanical properties. To accomplish this the processes of rolling of such shapes as sheets, or drawing of the wire, were carried out *while the metal was refrigerated to a desired temperature.*

TABLE IV.—EFFECT OF HOLDING AT SUB-ZERO TEMPERATURES ON IMPACT STRENGTH AT ROOM TEMPERATURE.[a]

Composition of steel: 0.06 per cent C, 9.80 per cent Ni 17.9 per cent Cr.

Heat Treatment: 1800 F., water quenched.

Samples: Charpy, standard keyhole notch.

Condition of Samples	Temperature of Testing	Impact Values, ft-lb.	Magnetism
As Quenched	Room	80 to 97	None
30 min. at −320 F.	Room	90 to 91	None
	−320 F.	71 to 73	None
6 months at −320 F.	Room	83 to 91	None
	−320 F.	72 to 75	None
12 months at −320 F.	Room	82 to 89	None
	−320 F.	73 to 77	None

[a] From T. N. Armstrong and A. J. Miller, "Notched Bar Impact Properties of Some Nickel Steels After One Year Exposure to Liquid Nitrogen," delivered at the National Conference on Petroleum Mechanical Engineering held by the American Society of Mechanical Engineers, Tulsa, Okla., October 7 to 9, 1946.

Processing Details:

Annealed sheets of the various types, subsequently rolled either at room[4] or sub-zero temperatures, were sheared into 1-in. wide strips. The strips were cut so that all rolling would be in the direction of the original rolling. The strips were cut into lengths which would yield sufficient material after rolling from which the tension specimens could be machined. All burrs were removed and the ends beveled to permit easy entry between the rolls. The thickness of the strips, which varied from 0.100 to 0.134 in., was measured and the thickness to which each was to be rolled was calculated.

The rolling was done on a two-roll mill, the rolls being approximately 5 in. in diameter and having a face of 6 in. During the rolling, the rolls were adjusted so that the thickness was reduced from 0.005 to 0.008 in. in each pass. During rolling at room temperature, no attempt was made to control the temperature of the specimens. This decision was deliberate in order not to deviate from established practice. The strips which were rolled at −105 F. and at −320 F. were placed in the liquid baths of the corresponding temperatures. After the strips had been cooled to the bath temperature (approximately 5 to 10 min.) they were quickly removed individually and quickly given one pass through the rolls. After measuring the thickness, they were again immersed in the bath and cooled to the rolling temperature. This process was repeated until the desired thickness was obtained. No attempt was made to extract the heat from the samples as they were being rolled because of the obvious complexity of the necessary equipment. Another reason was the possibility of damage to the rolls if the latter were cooled to these low temperatures. The most valid reason, however, was the realization that the heat developed within the sample during each pass would certainly exceed any effect from contact of chilled samples with unchilled rolls. It was believed that the temperature rise from the time the material was removed from the bath until it entered the rolls was not great, because each pass was completed within 10 sec. of the time the strip was removed from the bath. Comparative samples of the same gage (original) were rolled down in the same number of passes. Thus, while, admittedly, each rolling pass raised the temperature of the sample to an unknown degree, this circumstance accompanied rolling of each sample and the results are held to be comparable.

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[4] Rolling at room temperature was to provide a basis for comparison.

Two baths were used for cooling to −105 F.: one bath consisted of dry ice and alcohol, and the other was made up of dry ice, chloroform, and carbon tetrachloride. Liquid nitrogen was used as a cooling bath for the samples rolled at −320 F.

TABLE V.—EFFECT OF WIDTH ON ELONGATION.

Specimen	Stainless Steel Type and Processing	Gage Width, in.	Thickness, in.	Tensile Strength, psi.	Elongation in 2 in., per cent
No. 1	302—Rolled at room temperature	0.372	0.070	289 000	35.0
No. 2		0.374	0.070	291 800	37.0
No. 3		0.375	0.070	291 100	37.0
No. 4		0.376	0.070	290 500	36.5
No. 5		0.490	0.071	290 600	38.0
No. 6		0.490	0.071	288 600	37.5
No. 7		0.624	0.071	288 900	39.0
No. 8		0.624	0.071	290 200	38.0
No. 9	301—Rolled at room temperature	0.260	0.070	300 000	26.0
No. 10		0.385	0.070	302 000	27.0
No. 11	304—Rolled at room temperature	0.259	0.071	256 000	35.0
No. 12		0.385	0.071	257 000	39.0

As was summarized above, the tensile properties of ferrous alloys including stainless steels are considerably higher at sub-zero temperatures. This circumstance is responsible for the greater power required of the rolling equipment when rolling is done by first refrigerating the steel to the desired sub-zero temperature. In attempting this processing, therefore, proper attention must be given to the matter of required size and power of the rolls or other working equipment.

Test Specimen and Effect of Inherent Variables in Testing Technique:

The tension tests on steels in various conditions of processing were carried out, as shall be described below, at three temperatures, namely, room (about 78 F.), −105 and −320 F. For tests at room temperature, standard specimens of $\frac{1}{2}$-in. wide test sections were used. For the lower temperatures tension specimens, the width of the gage section had to be reduced to $\frac{3}{8}$ in. and in a few cases even less. This precaution was necessary in order to cause failure to occur within the gate length: the higher rate of strain hardening at sub-zero temperatures strengthened the $\frac{1}{2}$-in. wide gage section to such an extent that failure would occur in the shank because the latter, being at room temperature, was *weaker* than the reduced section. This necessary reduction in the gage section raised the question as to the possible effect of the width on the results of the tests, primarily elongation. To obtain factual data, a series of samples were machined to various widths of gage section. The tests were conducted at −320 F. and the results are summarized in Table V.

TABLE VI.—EFFECT OF SMALL CHANGES IN ROLLING TEMPERATURE.

Type	Heat	Cold Reduction and Temperature of Rolling	Tensile Strength (at −320 F.), psi.	Elongation in 2 in., per cent
301.......	2553	40%—Room temperature	320 000	22
301.......	2553	40%—212 F.	310 000	23
301.......	2672	40%—Room temperature	304 000	26
301.......	2672	40%—140 F.	297 000	27
301.......	2672	40%—212 F.	295 000	28

The above information furnishes definite proof that the test results secured on samples of gage width within the above limits (no smaller or wider specimens were used in this investigation) are strictly comparable.

As has already been mentioned, the rolling of certain specimens was done at "room temperature," that is, without prior refrigeration; these specimens were needed to furnish a basis of comparison between rolling at room and sub-zero temperatures. Since this experimental rolling was done at various times, the possibility of small changes in the temperatures of the samples caused by rolling could not be excluded. To ascertain the effect, if any, of small changes in rolling temperature, several specimens were rolled at 70 (room temperature), 140, and 212 F. The results obtained and reproduced in Table VI are considered normal for the conditions of the test. As would be expected, some change in the values for tensile strength or elongation was noted as the result of the above variable, but this change can be considered, as will be seen later, quite minor compared with that induced by sub-zero rolling.[5]

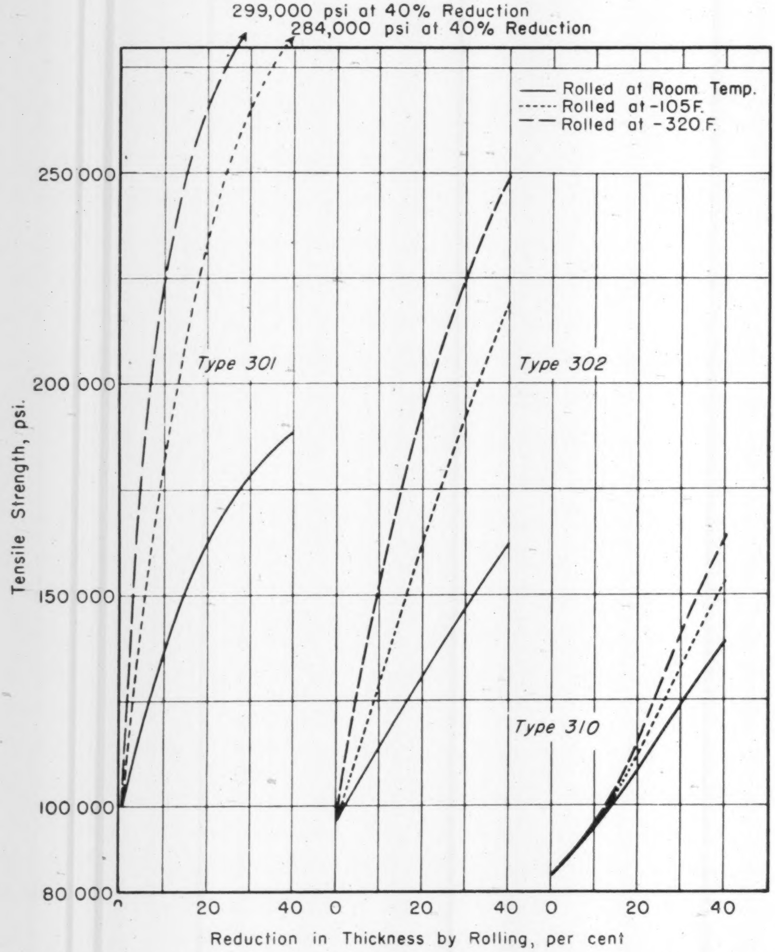

FIG. 3.—Effect of Rolling Temperatures on Tensile Strength of Types 301, 302, and 310 Stainless Steel.

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[5] An interesting paper by Lincoln and Mather (8) was made available since the start of this investigation. In this paper is recorded the same *trend* as in our work. However, our results show lesser influence of 140 and 212 F. than the results of Lincoln. It is possible that the difference in analysis of the studied steels are responsible for the lack of complete agreement on experimental values.

In some cases, considerable time elapsed between rolling and testing. The possibility that room temperature "aging" may be taking place had to be considered. In Table V, specimens Nos. 1 and 2 were tested within one week of rolling. Specimens Nos. 3 and 4 were held, deliberately, for two months before testing. Thus, it is shown that no aging took place at room temperature.

Effect of Rolling Temperature (from 70 F. down to −320 F.) on the Tensile Properties of Stainless Steels at Room Temperature:

Following the procedure of rolling at three different temperatures at which various samples were refrigerated, tension specimens were prepared and tests in tension were carried out at room temperature. These results have been assembled in tabular form.[6] The general conclusion, discussed later in detail, is this: the sub-zero temperatures of rolling have a marked effect on the ensuing mechanical (tensile) properties. The steels selected for testing, namely, commercial types 301, 302, 304, 310, and 347 represent various degrees of "austenite stability," a term already used. The improved mechanical properties obtained in these steels by mechanical rolling are governed by the rate of work hardening which is controlled by the chemical composition of the alloy in question—in other words, by the degree of stability of its austenite. This is, of course, well known from our experience with the rolling of these steels at room temperature. The stability of austenite in the case of any single stainless steel is decreased as the temperature is lowered, and it is for this reason that as mechanical working is applied at these sub-normal temperatures, the rate of hardening increases. It follows, therefore, that rolling the steels at sub-zero temperatures produces substantial increases in the tensile properties and hardness over those that are developed in sheet samples of similar steels by rolling to the same amount of reduction but at room temperature. The magnitude of increase depends on the initial stability of the austenite. The influence of temperature of rolling was much more pronounced in types 301, 302, and 304 than in type 310. It was also noted that although maximum increase was obtained when rolling was conducted at −320 F., the percentage improvement was greater in going from room temperature down to about −105 F. than from the latter down to −320 F. Figure 3 presents a summary of this observation on three types of stainless, steels of different degrees of austenite stability, namely, type 301, 302, and 310.

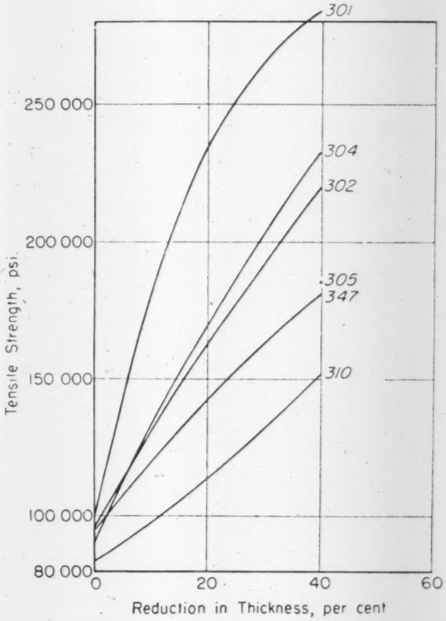

FIG. 4.—Comparison of Tensile Strengths Developed in Various Stainless Steel Grades by Rolling at −105 F.

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[6] This table contains a complete record of *all* tests discussed in this paper and is, therefore, quite voluminous. Since appropriate data from this table is presented in graphical form elsewhere in the paper, it seemed advisable not to include the table in the paper, thus saving considerable space. Copies of the table are available upon request to the authors.

In the case of type 301 stainless steel, rolling to 40 per cent reduction in thickness at room temperature (common practice) and at −320 F. resulted in an increase in tensile strength of this steel of about 50 per cent from about 200,000 psi. to 300,000 psi. Types 302 and 304 showed about the same increase, that is from 160,000 psi. to 250,000 psi. Type 310, when rolled the same amount and under the same rolling temperatures, increased in room tensile strength only from 140,000 psi. to about 165,000 psi.

Because the effect of sub-zero rolling is quite pronounced at −105 F., other steels were tested after rolling at this temperature. A summary of data on the level of strength that −105 F. rolling can develop in a number of common austenitic stainless steels is shown in Fig. 4. Here are recorded experimental values of tensile strengths in various stainless steels when the latter were rolled to 20 per cent and 40 per cent reduction which, as stated, was carried out at −105 F. Only one value was obtained for type 305 as can be seen from the figure.

In Fig. 5 are shown graphically *the differences* in tensile strength of various types of stainless steels when rolled with the same amount of reduction in thickness, one set at room temperature and the other at −320 F.

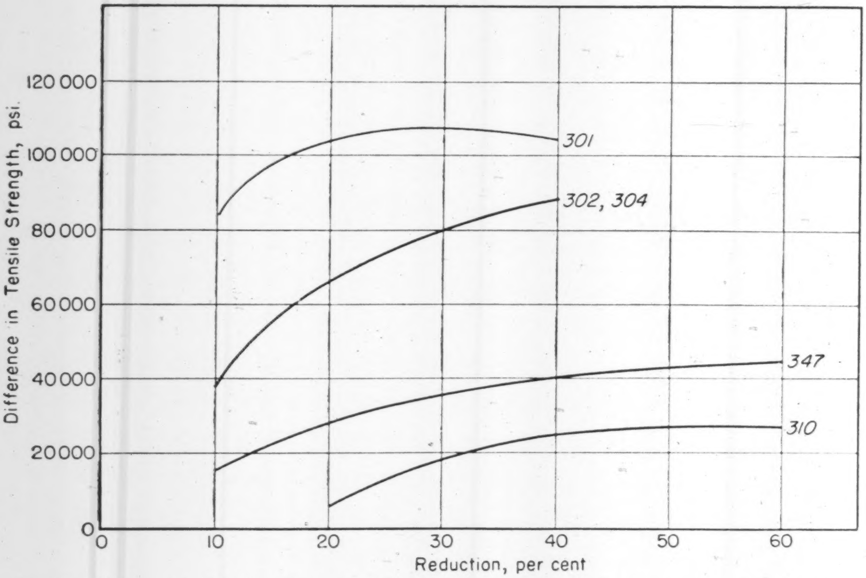

FIG. 5.—The Increase in Tensile Strength in Various Standard Stainless Steels Developed by Rolling at −320 F. as Compared to the Same Amount of Reduction at Room Temperature.

Higher levels of strength than cited above were obtained by rolling 60 per cent of reduction in thickness. With this heavy reduction, the practical limit of rolling for type 301 was approached. In fact, these samples were unsuitable for testing due to excessive edge cracking.

Numerous data on the yield strength, proportional limit, etc., obtainable by sub-zero processing have been assembled.[6]

The study of experimental values for proportional limit would indicate that only in types 301 and 302 one might expect noticeable improvement. More stable alloys, type 310 for example, showed negligible improvement. In view of the experimental difficulties associated with determination of proportional limit at sub-normal temperatures on sheet samples, the values for such properties are difficult to obtain with the desired accuracy. It seems wise to speak in terms of the "range" of elastic properties rather than individual values.

Modulus of elasticity is decreased when rolling is done below room temperature rolling, much in the same manner as in case of standard, room temperature rolling.

Although the improvement in strength as developed by the described rolling at sub-zero temperatures is of considerable interest, even more promising is the study of the effect of low-temperature rolling on the relative ductility of the investigated steels. Under standard practice of rolling, that is at room temperature, the high strength of stainless steels is accompanied by relatively low ductility—as would be expected.

In the case of sub-zero rolling the *increase* in ductility *for a given level of tensile strength* is made quite clear by the plot of experimental values for tensile strength and ductility *versus* percentage cold work as has been done in Fig. 6 in the case of type 302 steel.

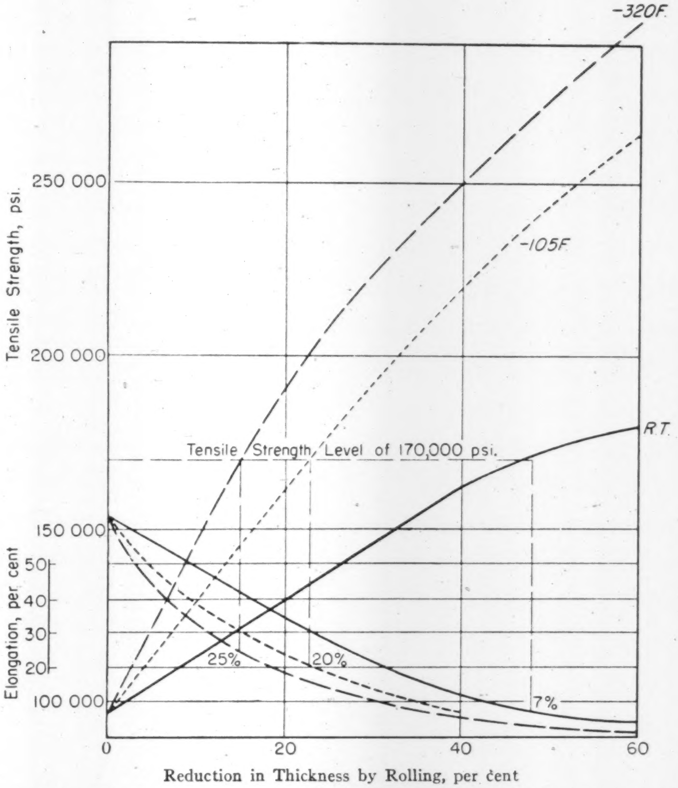

FIG. 6.—Tensile Strength and Elongation of Type 302 Stainless Steel Rolled at 70, −105 and −320 F., Tested at 70 F.

A commercial heat of type 302 of the typical analysis was rolled in the manner described above, that is at three different temperatures. The tensile strength and elongation developed at each stage of reduction in thickness were plotted.

TABLE VII.—EFFECT OF SUB-ZERO ROLLING.

Tensile Strength Level, 170,000 psi. (Arbitrarily Selected)				Correlation: Tensile Strength of 170,000 psi., Required			
Temperature	Reduction Required, per cent	Rolling Temperature	Corresponding Elongation, per cent	Temperature	Reduction, per cent	Rolling Temperature	Corresponding Elongation, per cent
Room	48	Room	7.0	Room	53	Room	6.0
−105 F.	23	−105 F.	20.0	−105 F.	25	−105 F.	15.0
−320 F.	15	−320 F.	25.0	−320 F.	18.3	−320 F.	22.0

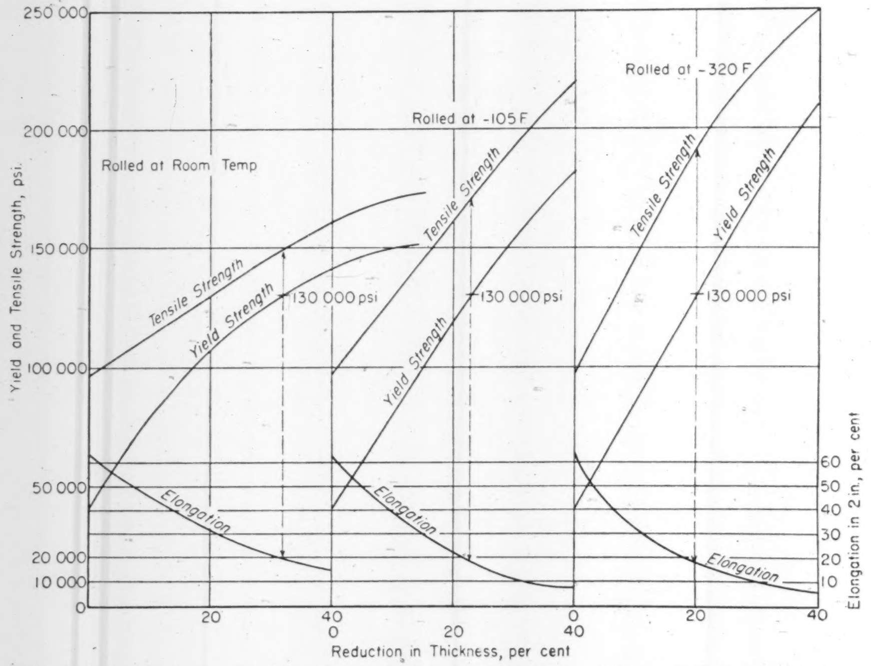

FIG. 7.—Relation Between Yield and Tensile Strengths of Type 302 Cold Rolled at Room and Sub-Zero Temperatures.

Let us assume, for example, that it is desired to have the product of about 170,000 psi. tensile strength. From Fig. 6 one can easily see that sub-zero rolling to the desired tensile strength produces a material with much improved ductility. Another observation, is that rolling to a desired strength takes much less reduction at sub-zero than at room temperature.

To make this conclusion quite clear, pertinent data are assembled in Table VII. In the left column of this table are found values for the elongation and per cent reduction in thickness taken from Fig. 6. The data under the heading "Correlation" were secured independently, while developing information on other properties of sub-zero rolled stainless steels. In the latter case a steel of the same type as 302, but of slightly different actual analysis was used. The approximate percentage of "required" reduction to produce a tensile strength of 170,000 psi. was read from Fig. 6. Then samples of the second steel were rolled to these reductions and subsequently tested to determine tensile strength and elongation. The two independent tests are obviously in good agreement. We accept these and other results as a satisfactory proof of our observation that at a given level of strength better ductility is provided by sub-zero working.

In many structural assemblies the stress calculations are based either on ultimate strength or on the yield strength of the material in question. Values for yield strength are, therefore, of considerable importance, and their determination in variously processed steels was included in this investigation.

In Fig. 7 are plotted the values for yield strength (average, 0.2 per cent offset), tensile strength, and elongation for type 302 stainless steel processed under various conditions, that is rolled a desired amount at room temperature, at −105 F. and at −320 F. Several interesting observations can be made. One of them is that the spread between yield strength and tensile strength in the case of rolling at room temperature is considerably smaller than when the steel is rolled either at −105 F. or −320 F. In other words, under conditions of sub-zero rolling the resultant product possesses a spread between yield strength and tensile strength that we usually associate with steels in which high strength values are induced not by cold rolling, but by heat treatment. Assuming that stress calculations will continue to be based *either* on tensile strength *or* yield strength equal to two-thirds the tensile strength, *whichever is the lowest,* the steels with the characteristics of those rolled, shall we say at −105 F., should be favored by the designer and stress analyst.

Let us assume for example that the design calls for yield strength of 130,000 psi. This value of yield strength corresponds to a tensile strength of 170,000 psi. of steel rolled at −105 F. and discussed in connection with Fig. 6. It is easy to see from the data in Fig. 7 that this yield strength corresponds to a tensile strength of about 149,000 psi. in the steel rolled at room temperature and to a tensile strength of 190,000 psi. in the steel rolled at −320 F. At the same time, and this is worthy of attention, the ductility of each steel remains practically the same. Table VIII summarizes the data.

TABLE VIII.—TYPE 302 STAINLESS STEEL ROLLED AS INDICATED.

Rolling Temperature	Arbitrarily Selected Yield Strength, psi.	Corresponding Tensile Strength, psi.	Elongation, per cent
Room	130 000	149 000	20
−105 F.	130 000	170 000	18
−320 F.	130 000	190 000	18

Detailed data on steel, type 304, of another analysis have been secured[6] which show that steel of this type behaves somewhat similarly to type 302; apparently the ratio between yield strength and tensile strength in types 301, 310, and 347 is influenced by the marked difference in the stability of the austenite in these steels.

The improvement in ductility as the result of the lowering of the rolling temperature takes place in types 302, 304, and 347—from among the investigated steels. It is our opinion that it would also be found in type 316. Only a few "spot" checks showed that we may expect it in type 305. The improvement *does not* take place—at least to a marked degree

—in the *more* stable grade such as type 310 and in the *least* stable grade such as 301, except, in the latter case, at fairly high levels of tensile strength. In the case of type 301 (at 230,000 psi. tensile strength) there appears to be an optimum in ductility at rolling temperatures close to −100 F.

A summary of information showing what ductility may be expected in several standard stainless steels rolled to arbitrarily selected values of tensile strength is given in Fig. 8. More specifically, one finds in the figure the elongation values at each of the working temperatures for tensile strengths of 160,000, 175,000, and 230,000 psi. for stainless steel types 301, 302, 304, 310, and 347.

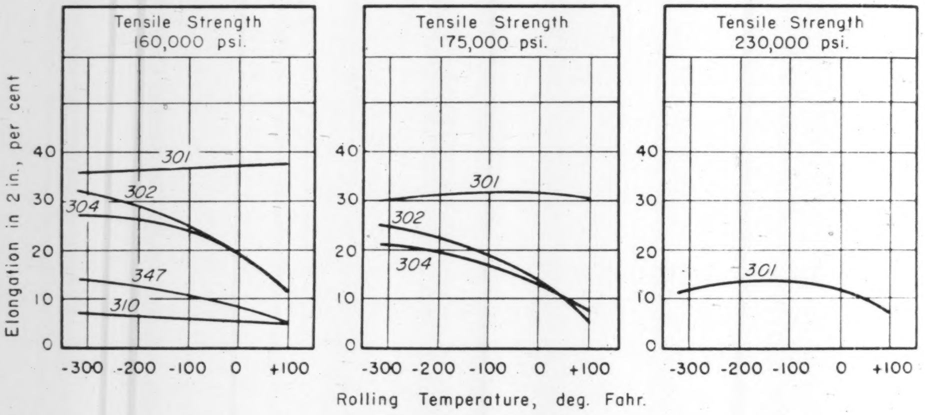

FIG. 8.—Effect of Rolling Temperature on Elongation of Various Types of Stainless Steels at Constant Tensile Strengths.

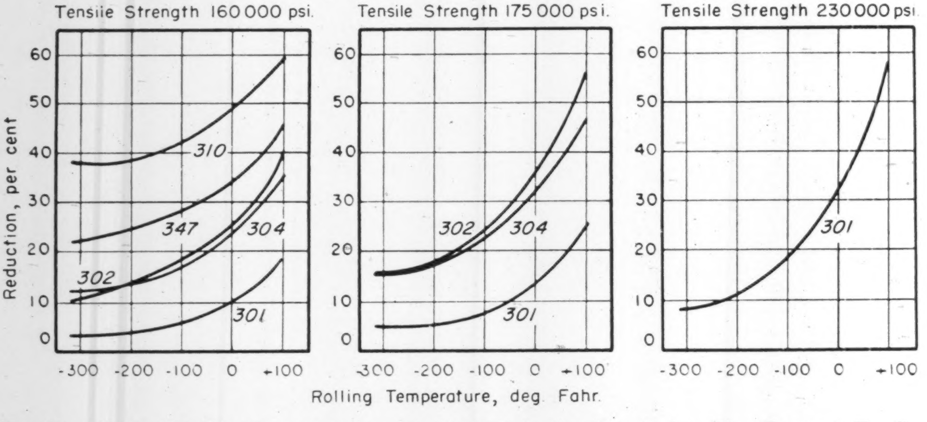

FIG. 9.—Effect of Rolling Temperature on Per cent Reduction Required to Give Constant Tensile Strengths.

As can be seen, the levels of ductility of the several stainless steels decrease as the austenite of such steels becomes more stable. This is due in part to the greater reductions necessary to produce a given tensile strength. However, it is believed that the more stable materials are apt to have comparatively lower ductilities even when given the same amount of cold reduction as the less stable steels, but at rolling temperatures which would result in equal tensile strength. Thus, rolling types 347 and 302 approximately 31 per cent at −55 F. and +35 F., respectively, would develop a tensile strength of 160,000 psi., but the more stable alloy type 347 would have an elongation of 11 per cent as compared with 17 per cent for type 302.

An important conclusion which we derived from the present studies, and which was already mentioned, should be emphasized. This is that as the temperature of rolling is lowered, the amount of reduction required to produce a given tensile strength is decreased and by quite a substantial amount. In certain operations, as for example, wire drawing, this observation might be of considerable importance.

Interestingly enough, all types of stainless steels which we investigated follow this rule *regardless* of the degree of stability of austenite. The summation of experimental data pertaining to this observation is shown in Fig. 9.

Effect of Sub-Zero Rolling on Hardness:

The values for hardness obtained through rolling at sub-zero temperatures are higher than the hardness of steel rolled at room temperature (with the same amount of reduction). A summary of the experimental data is given in Fig. 10, where the hardness values are compared on the basis of the same total amount of deformation. As in the case of the tensile properties, the increase in hardness is controlled by the stability of the austenite, that is the chemical composition of the alloys. It is to be realized, however, that in the case of austenitic stainless steels the hardness and tensile properties are not simply related. Rockwell "C" hardness up to 55, which was obtained by sub-zero rolling in types 301 and 302, cannot be obtained in these steels by ordinary rolling.

Effect of Stress-Relieving Treatment:

Previous work (9) established the beneficial effect of the so-called low-temperature heat treatment on the properties of austenitic stainless steels rolled at room temperature. It was logical, therefore, to inquire about the mechanical properties of steels rolled at sub-zero temperatures when such steels are given a subsequent stress relieving heat treatment. The obtained data are reproduced in Table IX.[7] In all (except a few cases of small amounts of rolling, that is less than 20 per cent) the yield strength values are improved. The improvement is influenced by the analysis of the steel and the processing. The difficulties associated with the determination of proportional limit are considerable, and while actual values may vary from test to test the results do show a real trend for an increase in proportional limit. Increase in tensile strength is to be noted. The effect on ductility is small; if anything, in a few cases the ductility seems to be slightly lowered.

It is felt that these results are indicative of what may be expected from the stress relieving of steels processed under various conditions of temperature and rolling reductions.

PROPERTIES AT SUB-ZERO TEMPERATURES OF VARIOUSLY PROCESSED STAINLESS STEELS

Introduction and Information on Procedure:

As was mentioned in the introduction to this report, considerable information has been published on the effect of testing temperatures, including those below normal, on the mechanical properties of austenitic stainless steels. Most of the

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[7] Additional data are discussed later in the paper under the heading "Effect of Prolonged Exposure at Elevated Temperatures on the Mechanical Properties at Room Temperature," see p. 926.

TABLE IX.—EFFECT OF SHORT-TIME AGING ON PROPERTIES AT ROOM TEMPERATURE.

Material	Conditions of Rolling[a]	Subsequent Heat Treatment	Proportional Limit, psi.	Yield Strength, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent
301.........	As received	None	18 000	34 000	102 000	72.0
	10% at R.T.	None	19 000	78 000	120 000	44.5
		550 F—24 hr.	24 000	86 000	119 500	46.5
	10% at −105 F.	None	21 000	84 000	153 000	32.5
		550 F.—24 hr.	24 000	86 000	142 000	40.0
	10% at −320 F.	550 F.—24 hr.	26 000	95 000	155 000	31
	20% at R.T.	None	21 000	96 000	139 500	32
		550 F.—24 hr.	26 000	117 000	146 000	33
	20% at −105 F.	None	22 000	128 000	206 000	18.5
		550 F.—24 hr.	45 000	159 000	193 500	15.5
	20% at −320 F.	None	33 000	182 000	236 000	10
		550 F.—24 hr.	42 000	218 000	241 000	5.5
	40% at R.T.	None	32 000	156 000	186 000	9
		550 F.—24 hr.	33 000	166 000	194 000	8.5
	40% at −105 F.	None	122 000	268 000	273 000	3.5
		550 F.—24 hr.	157 000	275 000	276 000	2
	40% at −320 F.	None	163 000	291 000	295 000	3.5
		550 F.—24 hr.	236 000	308 000	309 000	3
	60% at R.T.	None	45 000	209 000	236 000	3
		550 F.—24 hr.	59 000	216 000	242 000	2
	60% at −105 F.	None	172 000	311 000	318 000	1.5
		550 F.—24 hr.	185 000	313 000	316 000	1.5
302.........	As received	None	23 000	38 000	90 000	58
	14% at −105 F.	None	26 000	97 000	139 000	26
		550 F.—24 hr.	31 000	97 000	131 500	27
	20% at R.T.	None	30 000	105 000	124 500	27
		550 F.—24 hr.	33 000	102 000	121 000	28.5
	20% at −320 F.	None	27 000	124 000	189 000	15.5
		550 F.—24 hr.	34 000	143 000	186 000	11
	40% at R.T.	None	46 000	144 000	162 500	8.5
		550 F.—24 hr.	48 000	155 000	172 000	8.5
	40% at −105 F.	None	49 000	192 000	225 000	3.5
		550 F.—24 hr.	63 000	221 000	236 000	3.5
	40% at −320 F.	None	52 000	227 000	250 000	4
		550 F.—24 hr.	59 000	255 000	260 000	2.5
	60% at R.T.	None	57 000	166 000	189 000	3.5
		550 F.—24 hr.	58 000	182 000	202 000	3.5
	60% at −105 F.	None	75 000	259 000	264 000	1.5
		550 F.—24 hr.	97 000	273 000	280 000	1
	60% at −320 F.	None	99 000	295 000	299 000	1.5
		550 F.—24 hr.	100 000	306 000	307 000	1.5
304.........	60% at R.T.	None	58 000	161 500	192 000	5.0
		550 F.—2 hr.	68 000	182 000	200 500	3.0
	60% at −320 F.	None	57 500	273 000	278 000	1.5
		550 F.—2 hr.	55 500	286 000	297 000	1.5
347.........	60% at R.T.	None	35 000	141 000	169 000	3.5
		800 F.—1 hr.	56 500	160 000	177 000	5.0
	60% at −320 F.	None	68 500	202 000	214 000	3.0
		800 F.—1 hr.	52 500	217 000	292 000	2.0

[a] R.T. = room temperature.

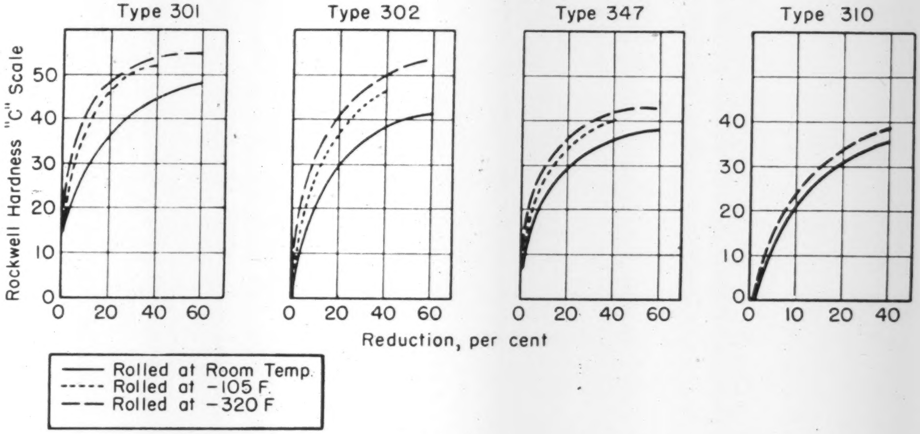

FIG. 10.—Per cent Total Reduction *versus* Hardness of Various Stainless Steels Rolled at Room and Sub-Zero Temperatures.

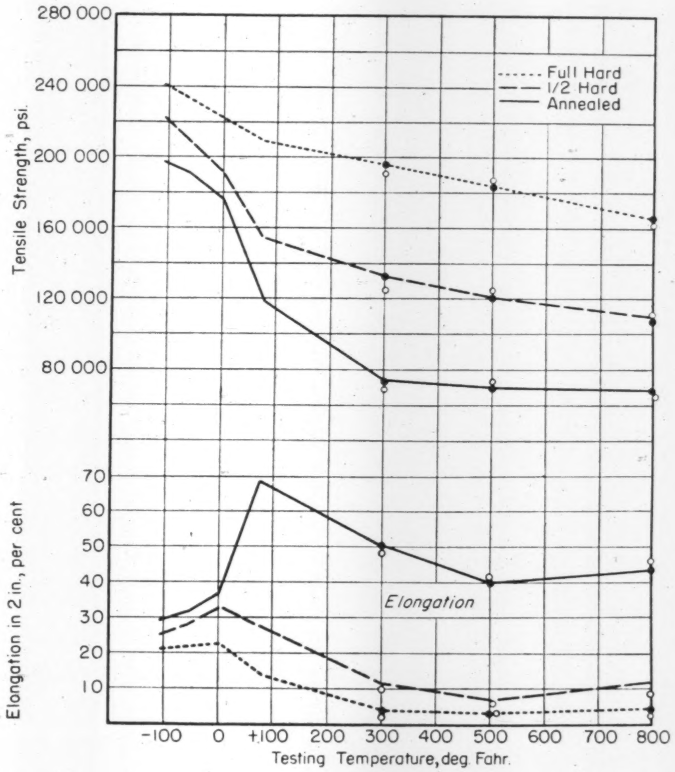

FIG. 11.—Tensile Properties of Annealed ½ Hard and Full Hard of Type 301 at Indicated Temperatures. (Holding Time at Temperatures: 1 hr. •, 100 hr. o).

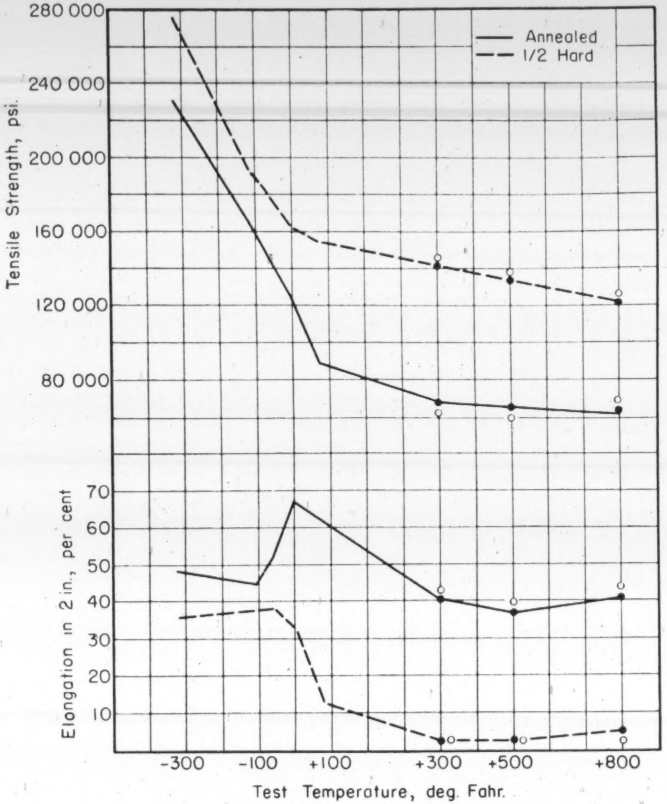

FIG. 12.—Tensile Properties of Annealed and ½ Hard Type 302 at Indicated Temperatures. (Holding Time at Temperatures: 1 hr. •, 100 hr. o).

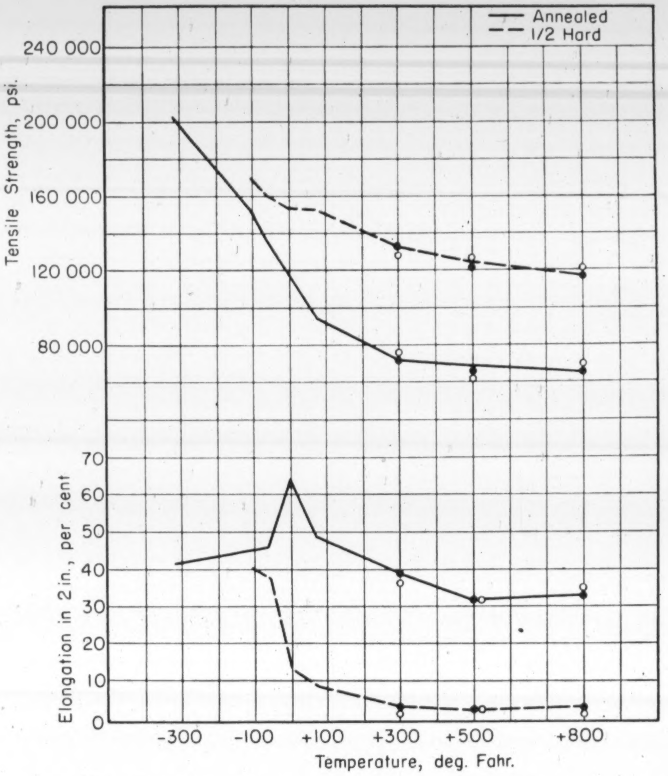

FIG. 13.—Tensile Properties of Type 347, Annealed and ½ Hard at Indicated Temperatures. (Holding Time: 1 hr. •, 100 hr. o).

available data, however, pertains to the steels after the latter were subjected to the quench-annealing heat treatment. The desire for additional information on the effect of testing temperatures below zero on the properties (more specifically ductility) of variously processed (cold-rolled) steels was the purpose of this part of the investigation.

As in the work described above, the steels selected were of a different degree of austenite stability, but all were selected from the available standard types of austenitic stainless steels.

Rolling of the steels was done at room temperature and at two sub-zero temperatures: −105 and −320 F. Most of the testing to determine the influence of test temperatures were carried out at −105 and −320 F. Occasional "spot checks" at −60 F. were made on commercially rolled steels. To ascertain the possibility of the influence of *time at temperature,* a number of tests were carried out after the samples were subjected to sub-zero temperatures for 1 hr. and 100 hr.

Tests to Determine the Effect of Temperature on Tensile Properties:

Complete and detailed data have been assembled but not reproduced here in detail.[6] To illustrate the general effect on tensile strength, the pertinent values are summarized (for type 301) in Fig. 11. It is to be noted that commercially cold-rolled grades, designated in the figure as ½ H and Full Hard[8] follow the general rule for ferrous alloys, namely, that tensile strength is increased as the temperature of the test is lowered. The same is true of stainless steel type 302 (Fig. 12) and type 347 (Fig. 13). It is to be noted, however, that in all three cases the relative (or percentage) increase in tensile strength at sub-zero temperatures as against that at room temperature is less in the case of cold-worked than annealed steels, and furthermore this increase seems to be less as the stability of the austenite becomes greater (compare, for example, the data for 301 and 302).

This observation was further checked by experimentally rolling in the laboratory of standard type 302 (at room temperature) at reductions of 20, 40, and 60 per cent of the original thickness.

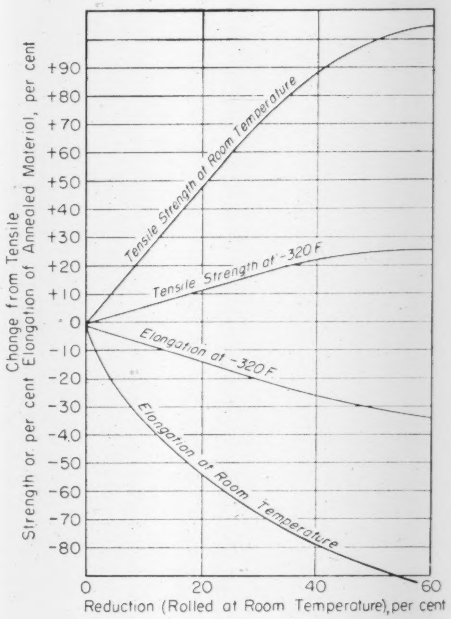

Fig. 14.—Per cent Change in Tensile Strength and Elongation from Annealed Material to that Rolled 20, 40, and 60 per cent and Tested Either at Room Temperature or −320 F.

These tension specimens were then tested at room temperature and at −320 F. In Fig. 14 are plotted the percentage *increase* in tensile strength and the percentage *decrease* in ductility as we go from annealed material to one with 20 per cent reduction, the second with 40 per cent reduction, etc. As can be seen, the effect of additional (greater) rolling is more pronounced under conditions of room temperature testing.

The same observation holds true in the

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[8] For definition of "half-hard," "full-hard," etc., see *Steel Products Manual,* Stainless and Heat Resisting Steels, Am. Iron Steel Inst., Section 24, p. 81.

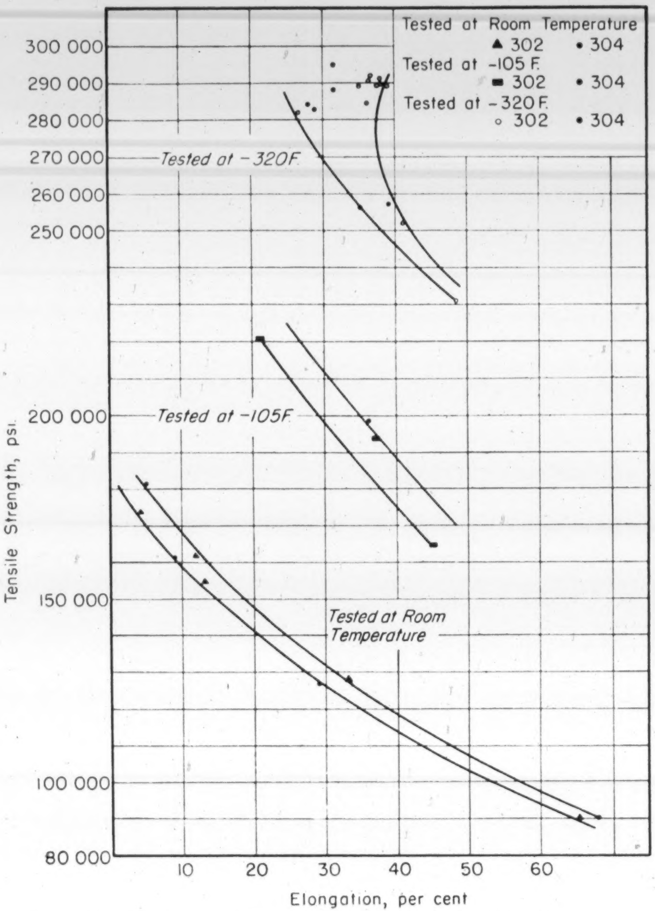

Fig. 15.—Summary of Experimental Values for Tensile Strength and Ductility of Annealed and Cold-Rolled Stainless Steel Tested at Various Temperatures.

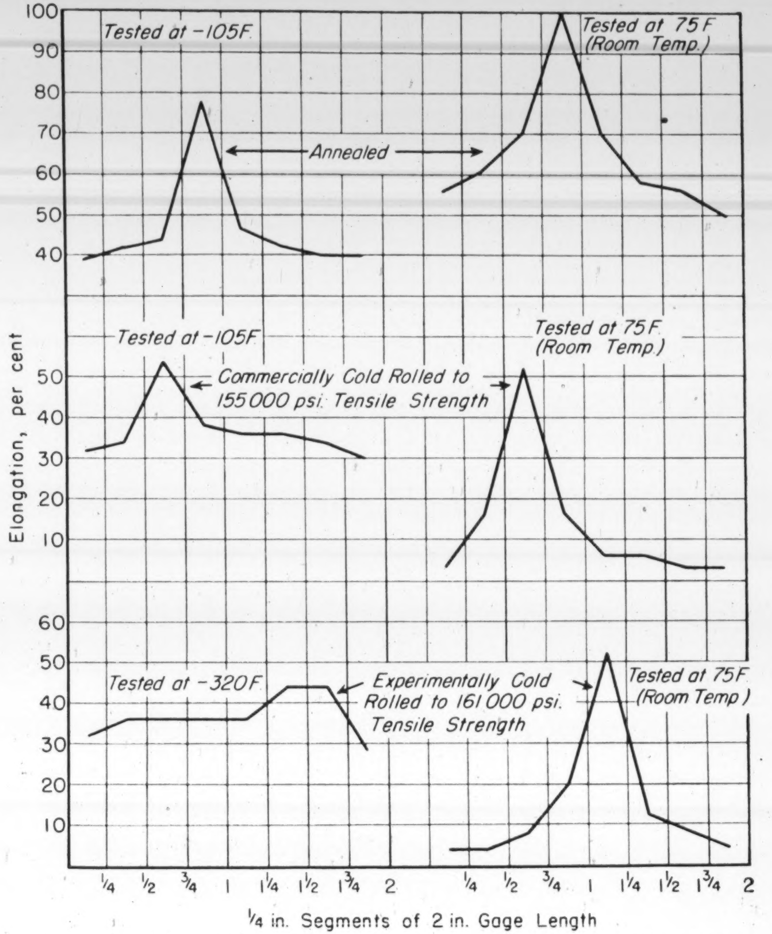

Fig. 16.—Effect of Testing Temperature on Distribution of Elongation in 2 in., for Annealed and Cold-Rolled Type 302.

case of type 347. In fact, limited data would indicate that the influence of the test temperature is even more pronounced. At room temperature the difference in tensile strength between annealed and $\frac{1}{2}$ hard steel is about 60,000 psi., but at −105 F. it is a bit under 20,000 psi. Thus, there would be relatively little advantage in the use of cold-rolled austenitic stainless steels at subzero temperatures if the purpose of such use was to provide high strength. Another and most interesting observation, however, must not be overlooked, namely, that ductility of the more stable cold-rolled types of stainless steels (such as 302 and 347) behaves in a manner totally unexpected in ferrous alloys: it (the ductility) actually goes *up* as the temperature of the test goes *down*. Furthermore, the increase in ductility is of very considerable magnitude, as can be readily seen from the data recorded on commercially rolled steels. Even quenched annealed steels seem to have optimum ductility at temperatures slightly lower than normal or room; shall we say between room and 0 F. Then, as the temperature is further lowered, these steels follow the usual pattern and the ductility goes down, still remaining, however, not too far below that possessed by the steels at room temperature.

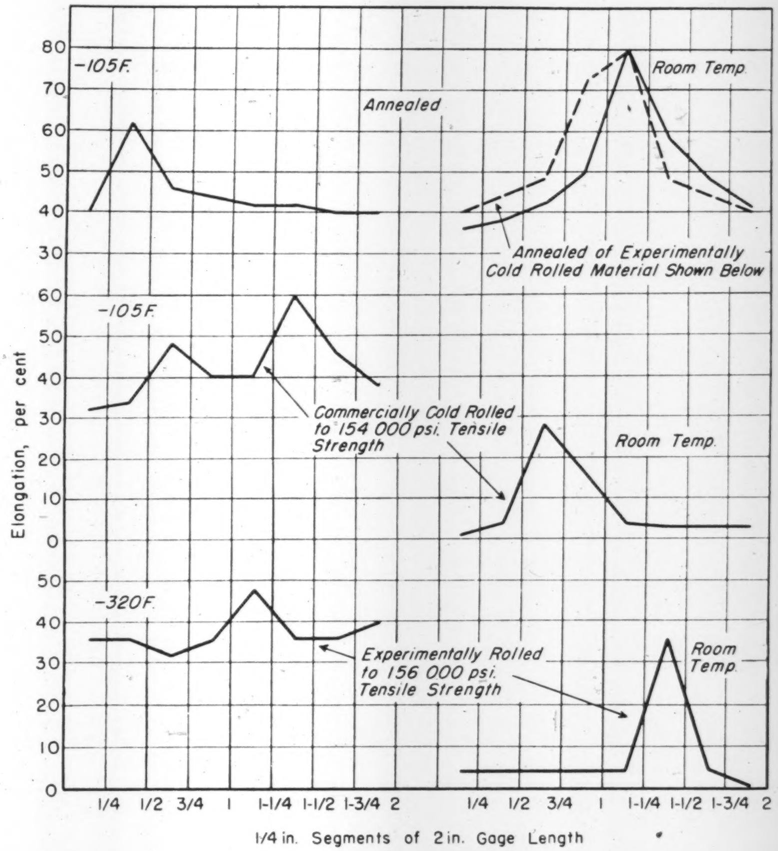

FIG. 17.—Distribution of Elongation in Type 347 Stainless Steel Tested at Various Temperatures.

An idea of what may be expected in

the way of mechanical properties of the more commonly used stainless steels (types 302 and 304) when such steels are tested at either room or sub-normal temperatures is presented in Fig. 15. In it are plotted the results of numerous tests for tensile strength and elongation of types 302 and 304, either annealed or cold rolled (in a standard manner at room temperature) and tested at −320 F. A few tests conducted at −105 F. are also included. It is to be seen that the experimental values plainly show the important combination of high strength and toughness as the tests are conducted at −105 and at −320 F. Although the full testing program was not carried out in the case of types 310 and 347, a few "spot checks" would enable us to predict the same behavior for these two types.

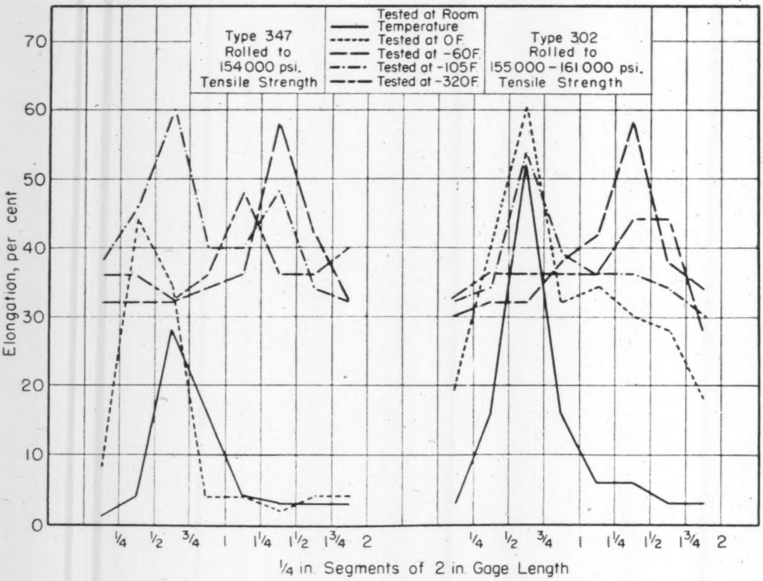

Fig. 18.—Distribution of Elongation of Cold-Worked Types 302 and 347 at Various Sub-Zero Temperatures.

Distribution of Elongation: Room Temperature versus Sub-Zero Testing:

A standard measure for ductility, namely, per cent elongation in 2-in. gage length, gives us *an average* stretch, the significance of which is obscured by the excessive stretch in the vicinity of the final break. During the war (10) and subsequently, considerable work was done on the *distribution* of elongation as measured over the standard 2-in. tension specimen, using as a unit of length (gage length) values of 0.25, 0.10 or even 0.01 in. In the present studies we used a gage length of 0.25 in., thus measuring the distribution of elongation over the 2-in. length specimens at eight points, 0.25 in. apart. Interesting observations are recorded in Figs. 16, 17, and 18 for types 302, 347, and 301 steels.

For the quench-annealed (soft) types 302 (Fig. 16) the pattern of elongation distribution is essentially the same at room and at sub-zero (−105 F.) temperatures. This pattern is that of a sharp maximum at or close to the final fracture which then gradually diminishes toward the ends of the gage length. Even at the ends of the 2-in. gage length the ductility is high, this ductility, however, dropping off when the steel is tested at −105 F.

In the case of *cold-worked steels*, however, the pattern of distribution of elongation is much altered by the temperature of the test. When the testing of such steels is done *at room temperature*, practically all elongation (stretch) is confined to that portion of the sample which contains the ultimate "break." Even a short distance away from the break the elongation is down to low values, obviously indicating that the location of the initial plastic "necking" determines the location of the final "break." At sub-zero temperature of testing (in this case −105 F., Fig. 16) we observe that maximum elongation at or near the "break" is the same as that in the sample tested at room temperature. But, a very considerable elongation—the actual measurements show 30 per cent or so—is found through the whole gage length of the tension test specimen.

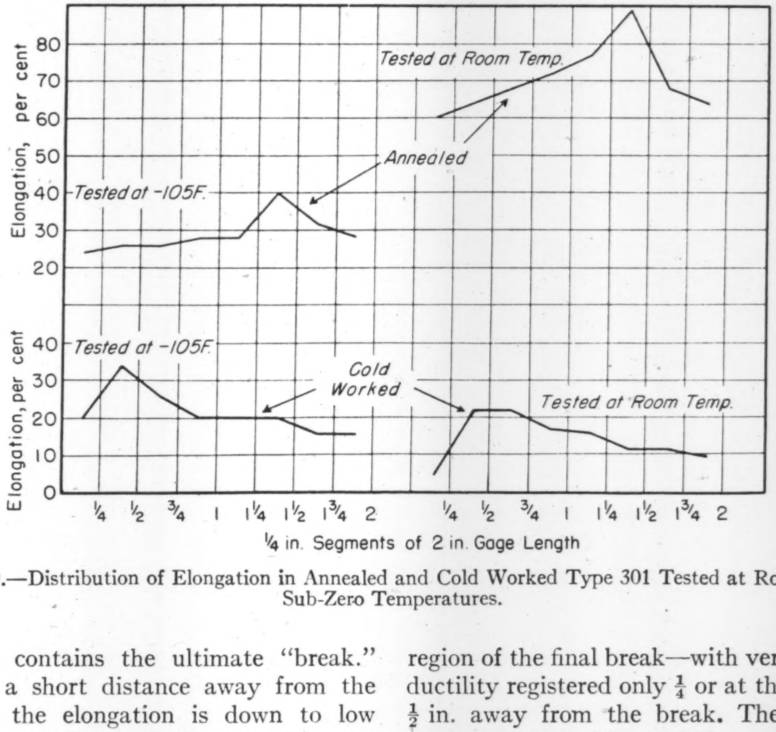

FIG. 19.—Distribution of Elongation in Annealed and Cold Worked Type 301 Tested at Room and Sub-Zero Temperatures.

Similar elongation patterns are also found in the case of type 347 (Fig. 17). Again, type 347 *cold rolled*, either experimentally in the research laboratories of The International Nickel Co., Inc. or by standard commercial methods, show that, when tested at room temperature, by far the largest proportion of the so-called ductility is confined to the region of the final break—with very poor ductility registered only $\frac{1}{4}$ or at the most $\frac{1}{2}$ in. away from the break. The same steels tested at either −105 or −320 F. show plentiful ductility distributed throughout the full gage length.

The results of additional studies, during which specimens of cold-rolled types 302 and 347 were tested at gradually decreasing temperatures, that is starting at room, then at 0, −60, −105 and finally at −320 F. (a different steel, but rolled to about the same tensile strength), are summarized in Fig. 18 and show how the distribution of elongation is affected by gradually lowered temperatures. In the case of type 302, it is evident that testing at 0 F. materially improves the ductility; further lowering of the test temperature to −60 F. merely tends to distribute the elongation more evenly throughout the whole gage length. It is interesting to note in this connection that the average per cent elongation in 2-in. gage length for tests at −60, −105, and −320 F. is for all practical purposes the same, namely, 38, 37.5, and 36 per cent.

In the case of type 347, however, lowering of the test temperature to 0 F. produces no alterations in the distribution, although greater "peak" is found at the break. A marked change is noted only when the temperature of the test is lowered still further, that is, to −60 F. Additional improvement seems to occur at −105 F. Thus, the optimum conditions for ductility and its distribution are obviously governed by the analysis of the steel. Undoubtedly, the amount of cold working (rolling) given to the steel prior to testing would also influence its characteristics, ductility-wise, at various sub-zero temperatures.

That the analysis of the steel has a marked influence is further illustrated by the studies of type 301. In the case of this steel, characterized by a high rate of work hardening, lowering of the test temperature injures the ductility of quench-annealed steels (Fig. 19). Further, the ductility of cold-rolled 301 is little improved by lowering of the test temperature from room to −105 F. Tests performed at a still lower temperature, namely, −320 F., show the *detrimental* effect of such low temperature on ductility.

Why Improved Ductility at Sub-Zero Temperatures?

The above described factual observations should be explained. It would be superfluous to state, merely, that at sub-zero temperatures "standard" elongation on 2-in. gage length is greater. In the first place it is not so in the case of the quench-annealed material. Secondly, not only the maximum elongation, that is at theoretical "zero gage length," is affected, but, as we described, the distribution of elongation in cold-rolled steels is altogether different. We must conclude that lowering of the test temperature affects in some manner the stretching process. Certain postulations will be necessary. The simplest explanation of improved ductility at sub-zero temperatures and, what is more important, its distribution, can be based on the effect of low temperatures on work hardening during plastic deformation.

It is quite possible that uniform elongation on the stress-strain curve (flow curve) is affected by the difference in the magnitude of strain hardening due to the difference in the temperature of the tests—room and below zero. This can be ascertained from a comparison of the true stress-strain curves, one conducted at normal (room) and the other at sub-zero temperature. It is believed, however, that it is the behavior of the metal as the necking-down point is approached that determines the resultant elongation and its distribution. The point where "necking" begins is, for all practical purposes, that point on the stress-strain curve where the effect of strain hardening, or more precisely, its rate becomes equal to the rate at which the true stress is increased, since the area in this specific location on the specimen is being continually reduced due to flow (deformation) under constant load. This should be visualized as follows: let us assume that at the moment of the beginning of necking the constant load is P and the area A. The stress is $\frac{P}{A}$ psi. As soon as even fractional necking takes place, the area becomes $A\text{-}a$ and the stress $\frac{P}{A\text{-}a}$, where $\frac{P}{A\text{-}a}$ is, of course greater than $\frac{P}{A}$. But at the same time resistance to deformation (through strain hardening which took place in the plastic range) is increased from S to S_1. If $S_1 - S$ is greater than $\frac{P}{A} - \frac{P}{A\text{-}a}$, then the deformation of the tensile test specimen will continue to be *uniform*, because strain hardening is more than compensating for the increase in stress caused by reduction of area at the point of necking. This is the first alternative. If the reverse is true, that is, the increase in stress due to reduced cross-sectional area is greater than the resistance to further deformation brought on by strain hardening (second alternative), the deformation will be confined locally, and will exhibit a sharp peak at the location of initial necking and final break. In the latter case, to state it somewhat differently, strain hardening of the material is not powerful enough to counteract the increased applied stress.

It is established that strain hardening of austenitic stainless steels by mechanical means is accompanied by transformation to ferrite and proceeds at a gradually lowered rate. This is illustrated, for example, by the fact that the relation of tensile strength *versus* amount of cold working (per cent reduction in total thickness) is not a straight line but one with a gradual decrease in slope. Thus, when samples of cold-rolled, that is already strain-hardened, stainless steels are tested at room temperature *further* strain hardening due to the just described deformation of the test sample corresponds to the second alternative, that is this strain hardening *is not sufficient to stop local deformation*, the cause of which is decreased area and thus increased stress. One would expect precisely the kind of distribution of deformation as we find in the case of cold-rolled stainless steels tested at room temperature.

Previous discussion and numerous data presented at the beginning of this paper show that the rate of strain hardening and accompanying ferrite formation[9] is *greater* when straining by mechanical means is carried out at certain low temperature ranges, the range depending on the composition of the steel. In type 302, for example, the strain hardening at −320 F. is about twice as great as at room temperature. Thus, the cold-rolled specimens tested at −320 F. might or should behave in accordance with the first alternative—as indeed the results indicate they do. Deformation continues to be quite uniform and high until the strain hardening ability, even at sub-zero temperatures, is exhausted, and any further elongation occurs locally until final breaking of the sample.

It is interesting to study in this connection Fig. 18 in which are summarized the results of deformation distribution and deformation "at the break" at five temperatures, from room down to −320 F. for types 302 and 347. Note two different patterns to be found within certain ranges of temperature. The local elongations (at the break) are quite comparable when type 347 is tested at room or at 0 F. A sharp change occurs in the pattern of distribution as the temperature of the test is further lowered. Much the same observation is evident in the case of type 302.

Of much interest to note in connection with the above studies is that the uniform elongation is directly applied in evaluating the forming characteristics of the steels. On the basis of our observations it could be predicted that cold-rolled stainless steels when refrigerated to the proper degree would form better under certain but not all forming processes, such for example as bending, dimpling or stretch forming. This was actually found to be the case. An illustration of it will be found in Fig. 20.

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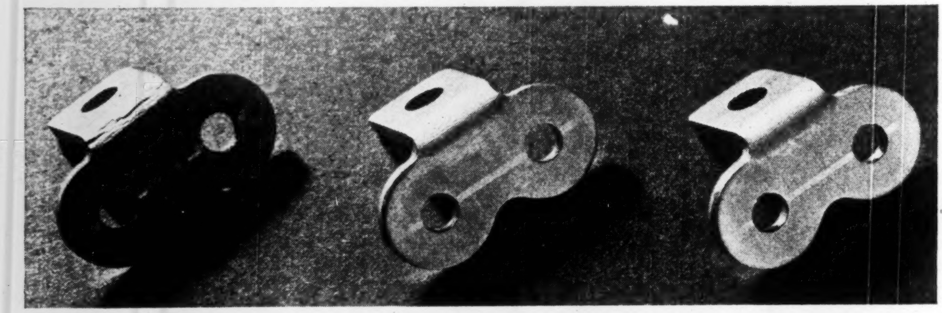
[9] Magnetic measurements, proving this statement, will be presented in the future. As yet, they are incomplete.

The patterns of deformation distribution in the case of annealed steels tested at room or sub-zero temperatures are alike, except for actual values. That this should be so is reasonable. When annealed steels are tested at sub-zero temperatures they exhibit such high strength that certain lowering of ductility is to be expected.



Sample No. 1.
Bent at 775 to 800 F.

Sample No. 2
Bent at room temperature.

Sample No. 3
Bent at temperature of dry ice.

FIG. 20.—Bending of Cold-Rolled Stainless Steel at Various Temperatures.

The observed difference in the distribution of elongation may also be explained as follows: There are two fundamental properties (or characteristics) in austenitic type stainless steels that tend to change with temperature. It is the relative change in these two characteristics that determine primarily the ductility. One characteristic is the rate of work hardening which affects the resistance of a material to slip. The other is a fundamental concept of cohesive strength or resistance to rupture. The rate of work hardening is the increase in strength as a result of a uniform deformation and is generally well described by the true stress-strain curve. The idea of its extent can also be given by the spread between the yield strength and tensile strength. The cohesive strength of a material is approximated by the true breaking stress, commonly referred to as breaking strength. Sheet specimens used throughout this investigation are not easily adapted to measure breaking strength. In order to do so, material in the form of rod was tested (the results are included with the other detailed data).[6] The relative change in the rate of work hardening and in the cohesive strength as well as the actual values may be used to explain the behavior of a material in regard to ductility in the light of the experimental evidence presented above. The data show that the breaking strength increases materially at sub-atmospheric temperatures and decreases rapidly immediately above atmospheric temperatures. The rate of change is approximately the same whether the steel is in the quench-annealed or cold-rolled condition. In the case of work-hardening of annealed material, however, there is a strong tendency for it to increase at sub-atmospheric temperatures with the actual amount varying widely from one material to another, and at elevated temperatures it is decreased, but not at a rapid rate within the range studied. The relative change of these two properties may account for the changes in ductility of any particular alloy and any particular temper. Work hardening, as was already stated, increases resistance to slip and should, therefore, decrease ductility as measured by elongation, but an increase in breaking strength counteracts this tendency by resisting fracture until further elongation has taken place. This means in its most simple concept that if the rate of work hardening is high in relation to cohesive strength of the metal (breaking strength), the elongation will be low while if the opposite is true the elongation will be high. These factors of cohesive strength and rate of work hardening are both a function of composition and temperature as can be shown by a close examination of the changes in ductility of all materials tested.

In annealed materials, the work-hardening factor is dominant at both room and sub-atmospheric temperatures, which is borne out by the very rapid increase in tensile strength that has been observed. It is to be remembered from previous discussions that when mechanical working and low temperatures are simultaneous factors the *rate* of work hardening is much increased. It would also be expected to be more pronounced in alloys of low austenite stability, since transformation to ferrite should in effect cause an increase in the rate of work hardening. It will be noticed that this is exactly the case. Type 301 annealed stainless steel showed the largest loss of ductility, and this loss was observed at temperatures immediately below room temperature. The pattern of distribution is practically unchanged as the temperature of the test is lowered, indicating again the predominant influence of work hardening. With type 302 quench-annealed sheet material, as well as type 347, both more stable austenitic alloys, there was first an increase in ductility in lowering the temperature to 0 F., and then a decrease (Figs. 12 and 13). Distribution of elongation at 0 F. is much more even. This would indicate that greater stability of the alloys allow the increased cohesive strength (induced by decreasing the temperature to 0 F.) to be the dominant factor. As the temperatures of the test are further decreased, the work hardening factor again becomes greater with a resulting decrease in ductility.

The cold-rolled or "hard" stainless steels are worked in processing, and when tested at room temperature show, therefore, a relatively low degree of ductility. A circumstance also to be remembered is the fact that their ability to be further work hardened (while being tested) is reduced. In fact, there is a definite limit to the amount of hardening that can be effected by working in the case of stainless steel of any particular composition. Obviously this limit is lower in relatively more stable austenitic materials than in those which readily undergo austenite-ferrite-carbide transformation on working. It can be reasoned, therefore, that the results of the testing of cold-worked stainless steels at sub-atmospheric temperatures are affected not so much by the rate of work hardening, which is limited, but by the fact that at sub-zero temperatures the cohesive strength (breaking strength) increases very rapidly. This increase in cohesive strength, we believe, causes the cold-rolled steels to resist fracture until considerably greater plastic deformation throughout the whole gage length has taken place than would be possible at room temperature. The more stable is the austenite in the general type of 18 per cent chromium—8 per cent nickel steels and the greater the previous cold working, the more effective should be the tendency to show relatively increased ductility at low temperatures. This is exactly what has been observed. The hard tempers of type 302, whether tested in the form of rod or sheet, and also type 347 displayed much increase in ductility at sub-atmospheric temperatures. Type 301, cold rolled to the so-called full hard temper, shows some little improvement in ductility. Less cold-rolled stock of this same steel showed practically no improvement. If we recollect the large work-hardening capacity of type 301, especially at sub-zero temperatures, and if we accept that the index of work hardening is indicated by the spread between yield strength and tensile strength, then it will be observed that correlation between this index and the changes in ductility is quite good.

MECHANICAL PROPERTIES OF QUENCH-ANNEALED, COLD-WORKED AND SUB-ZERO-ROLLED STAINLESS STEELS AT ELEVATED TEMPERATURES

A number of applications of stainless steels, both in the quench-annealed and cold-worked conditions, call for service within the temperature range from about 300 F. to 700 F. or even 800 F. In some of these applications the conditions of service are intermittent, that is the installation or part of it will be at temperature a relatively short time and then cool off to normal or air temperature. For example, certain parts near a power plant of an airplane represent such service conditions. What would be the effect of such intermittent exposure, different as to the time of exposure at elevated temperatures, on the properties of stainless steels when such properties are determined either at the temperature of exposure or after cooling down to normal?

To gain at least partial information, samples of types 301, 302, and 347 steels available in the quench-annealed, cold-worked (rolled at room temperature) and sub-zero-rolled conditions were held at temperatures of 300 F., 500 F., 800 F. or 1000 F. for either 1, 100 or 500 hr. and tested at corresponding temperatures. In addition, test pieces from the same steels were cooled rapidly to and tested at *room* temperature, to ascertain if any changes in the properties of the steels have been induced by such *relatively* long exposures as 100 and 500 hr. to all temperatures previously mentioned.

Tests at Temperature—Mechanical Properties After One Hour of Holding Time:

Examination of the results for type 301 (Fig. 11) shows that for all three tempers (annealed, half hard, and full hard)[8] there is a continuous decline in tensile strength with increasing temperature. Interestingly, the most pronounced effect of increased temperature is noted in the annealed material in the range of temperatures between 75 and 300 F. In fact, the effect of temperature in the range from 300 to 800 F. is practically negligible in the annealed material. The more cold-worked tempers showed much less influence of temperature from 75 to 300 F. The decrease in tensile strength of the cold-worked tempers appears to be more or less linear with increasing temperature (from 75 to 800 F.).

The yield strength,[10] which is truly more indicative of the load-carrying ability of the material, is less affected by temperature but does not behave in the same manner in all three tempers. The annealed material shows a gradual decrease with increasing temperature, which is the expected trend. With the harder tempers the decrease in yield strength with increasing temperature is greater the greater the amount of previous cold work. This is, also, we believe, as it should be.

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[10] The experimentally determined values for yield strength *are not* plotted in Figs. 11 to 13 (in order not to confuse the reader). These values are available in the detailed data referred to in footnote 6.

The lowering of ductility as indicated by elongation of the "as received," that is quench-annealed, materials is contrary to what one would expect and is difficult to explain. From previous observations, it is seen that the annealed stock displays a *decrease* in ductility at temperatures both above and below some optimum temperature.

The low ductility at elevated temperatures would seem paradoxical in view of the lower strength that accompanies it. The suggestion which at once comes to mind would be that work hardening during testing at elevated temperatures is considerably lowered and, thus, the increment $\left(\frac{P}{A-a} - \frac{P}{A}\right)$ psi., is greater than (S_1—S) psi., with the final result that we encounter "localized" failure rather than one which is preceded by a continuous slip. The low ductility of this steel at temperatures up to 800 F. (the lower limit of this range is difficult to place, but it certainly is at about 300 F. or lower) cannot be explained on the basis of structural changes, such as aging. The results of tests clearly show that the change in ductility due to temperature is not permanent, and lowering of such ductility is obliterated if the steels are again brought down to about room temperature and tested.

In the case of type 302, only annealed and half-hard materials were tested. A full hard temper of this type of stainless steel is not too well suited to fabrication processes, and the use of this temper in structural applications is rather restricted.

The results of the tests have been tabulated[6] and are shown in Fig. 12. It is shown again that in the annealed material the rather sharp drop in tensile strength occurs when the temperature is raised from about 75 F. to 300 F. Therefrom and up to 800 F. the lowering is considerably less, thus confirming the findings in the case of type 301. The values for yield strength are roughly parallel to those for tensile strength. In the case of half-hard tempers of this type of stainless, the drop in tensile strength and in yield strength with temperature is more regular, being, however, more pronounced as the temperature is increased than in the annealed grade. A rather high ratio of yield strength to tensile strength in the half-hard temper should be noted.

The ductility of either annealed or half-hard material suffers severe lowering between 75 and 300 F., but further increase in temperature had little effect. A slight recovery, which should be anticipated, appears to occur in the neighborhood of 800 F.

Substantially the same results as above were obtained during studies of type 347, annealed and half hard. Again one should note the sharp lowering in tensile strength as the temperature of the test is raised to 300 F. (Fig. 13). At temperatures above 300 F. and up to 800 F., the decrease in tensile strength is much more gradual. The experimental values for yield strength behave in the normal, expected way—again they are roughly parallel to those for tensile strength.

Ductility is again observed to drop off with increasing temperature. This drop is very pronounced in the case of the half-hard temper. As was already pointed out, this lowering in ductility is the effect of the test temperature and *not* of any permanent change in the material.

Effect of Longer Holding Time (at Elevated Temperatures) on Mechanical Properties:

As was previously mentioned, one of the important considerations in design for service at medium-elevated temperatues is the ability of a material to maintain its properties at these temperatures. In the present experiments, the holding time was extended to 100 hr., and in further studies, to be described sepa-

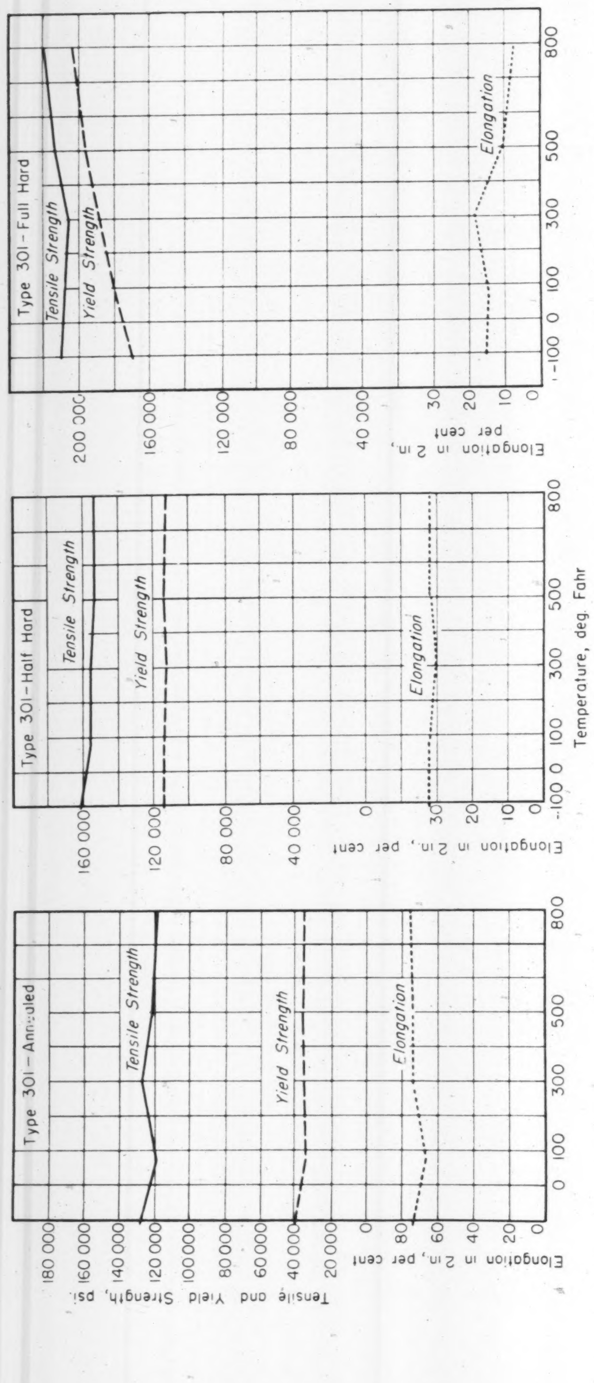

FIG. 21.—Results of Tests at Room Temperatures on Commercial Annealed and Cold-Rolled Stainless Steel After Holding for 100 hr. at Indicated Temperatures.

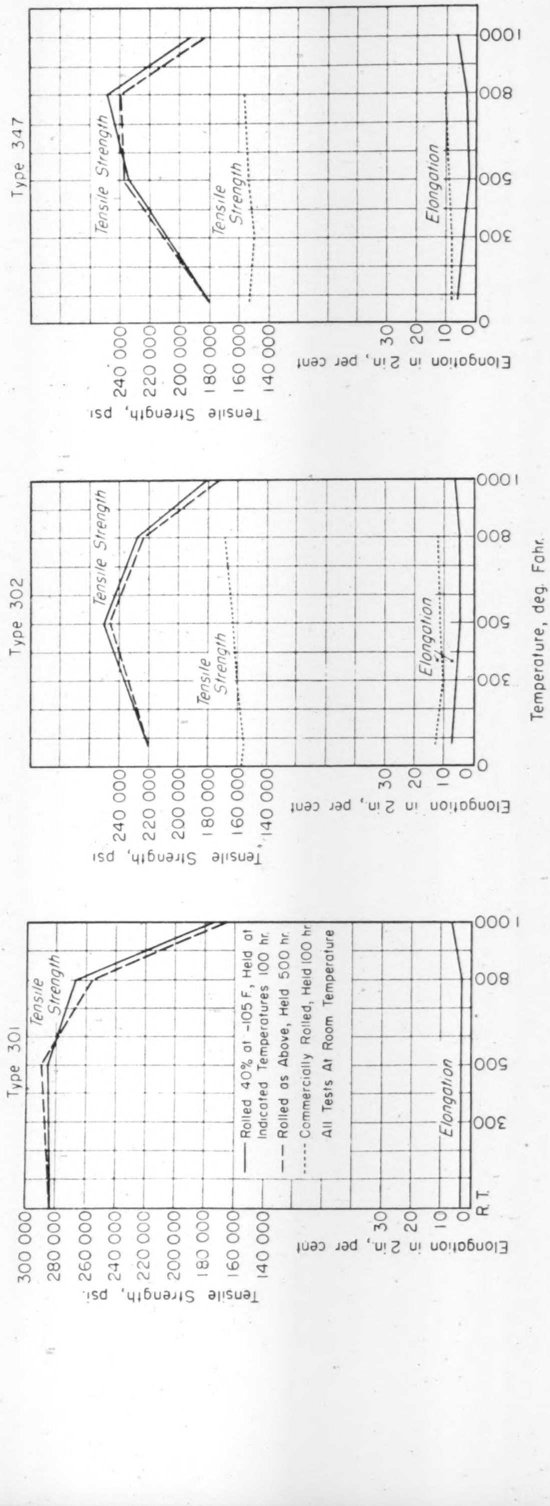

FIG. 22.—Effect of Elevated Temperatures on Properties at Room Temperature.

rately, to 500 hr. It is believed that the stated durations of exposure to elevated temperatures should give at least an indication of the changes that we may expect.

TABLE X.—EFFECT OF LONG-TIME AGING ON PROPERTIES AT ROOM TEMPERATURE.
All material rolled 40 per cent at −105 F.

Type	Aging Temperature, deg. Fahr.	Aging Time, hr.	Elongation in 2 in., per cent	Tensile Strength, psi.
301.........	None	None	4.0	275 000
	None	None	4.0	274 500
	500	100	1.0[a]	285 000
	500	100	3.5	287 500
	500	500	3.75	290 000
	500	500	2.5	290 000
	800	100	2.25	273 000
	800	100	3.25	260 000
	800	500	6.0	258 500
	800	500	5.0	249 000
	1000	100	5.0[a]	170 000
	1000	100	6.5	176 000
	1000	500	2.0[b]	170 000
	1000	500	6.75	160 000
302.........	None	None	5.5	235 400
	None	None	6.0	234 900
	500	100	4.5	255 000
	500	100	1.5[a]	245 000
	500	500	3.0	245 000
	500	500	3.0	250 000
	800	100	5.0	228 500
	800	100	5.0	227 000
	800	500	4.5	220 000
	800	500	4.5	225 000
	1000	100	8.0	179 000
	1000	100	5.0	181 000
	1000	500	4.0[a]	169 400
	1000	500	8.5	172 000
347........	None	None	6.5	189 500
	None	None	6.0	189 900
	500	100	—[b]	245 000
	500	100	2.5	235 000
	500	500	3.0	235 000
	500	500	2.5	236 500
	800	100	3.75	250 000
	800	100	3.1	247 500
	800	500	5.5	239 000
	800	500	3.75	240 000
	1000	100	7.0	195 000
	1000	100	6.25	189 000
	1000	500	2.0[a]	183 000
	1000	500	6.75	182 000

[a] Broke at punch mark.
[b] Broke outside punch mark.

The results of the tests, in detail, are to be found in Table X.[6] The graphic summary is represented by Figs. 11, 12, and 13.

The results were quite interesting and encouraging. It is evident from the data that holding for as long as 100 or 500 hr. at 300, 500, and 800 F. does not change, appreciably, the tensile strength, yield strength and indices of ductility as compared with values of only one hour holding time.

Effect of Prolonged Exposure at Elevated Temperatures on the Mechanical Properties at Room Temperature:

A summary of the tests will be found in Table X. Most pertinent conclusions are presented in graphical form in Figs. 21 and 22. In these figures a comparison is made between the properties of steels as rolled (plotted as "held at room temperature") and properties of the same steels of the same steels after 100 or 500 hr. at indicated temperatures.

Type 301, commercially annealed or cold rolled to half-hard temper, possesses properties at room temperature which apparently have not been adversely affected by holding for 100 hr. at temperatures up to and including 800 F. As will be seen from Fig. 21, the values for strength and ductility are the same for all conditions of previous histories. The same type 301 in the full-hard temper, however, seemed to have experienced a slight increase in strength with corresponding decrease in ductility.

The results of similar tests on type 301 that has been rolled in the research laboratories at sub-zero temperatures (Fig. 22) (consequently possessing considerably higher tensile properties) show that such properties are fully maintained after exposures, for as long as 500 hr., to temperatures up to and including 500 F. Holding for the same length of time at the next experimental temperature, namely, 800 F., resulted in a noticeable but not excessive lowering in the tensile strength.

Type 302, commercially processed, showed a steady but slight increase in room temperature properties as the heating was carried out at temperatures up to 800 F. (Fig. 22). Experimentally processed steels (sub-zero rolling to a considerably higher tensile strength) show a decided improvement in tensile properties after heating in the vicinity of 500 F. which is then followed by a gradual decline as the temperature is raised to 800 F. Even after heating at the latter temperature, however, the properties of the steels are substantially the same as if no long-time heating was applied. Obviously, heating the samples to and holding them at the above named temperatures is an "aging" treatment. Thus, it would appear that these steels do not suffer from "over-aging" during exposure of up to 500 hr. duration.

The observation that steel of this type processed originally to approximately 160,000 psi. tensile strength behaved differently from the one processed to a tensile strength of 220,000 psi. may be explained by the latter's higher tensile strength. It has been observed before that the higher the original tensile strength the lower is the temperature at which partial softening takes place.

Quite in line with previous observations, a further increase in the temperature beyond 800 F. has a marked effect. As is seen from Fig. 22, the strength falls off and the ductility begins to improve, indicating that partial annealing began to take place.

Type 347, commercially rolled, maintains its properties up to and including 800 F. for the time of heating of 100 hr. experimentally (sub-zero) rolled steel of the same type showed a continuous increase in tensile strength as the temperatures of heating were maintained up to and including 800 F. Again, this is attributed to the "aging" effect. This increase is of very substantial magnitude and is consequently of considerable interest. Beyond 800 F. partial softening begins to take place.

All of the above observations based on experimental results allow this general statement: Heating times of up to 500 hr. at temperatures as high as 800 F. (the temperature depends on the type of steel) have, apparently, no detrimental effects on the room temperature properties.

CONCLUSIONS

1. Austenitic stainless steels, annealed and cold worked, when held as long as 100 hr. at temperatures ranging from 0 to −320 F. do not undergo changes in mechanical properties at room temperature.

2. Mechanical working of steels *while the latter are refrigerated to temperatures as low as −320 F.* induces in these steels considerably greater rate of work hardening, with a corresponding increase in tensile strength, yield strength and hardness as compared with steels processed in a standard manner, that is, at room temperature.

3. At a given value of tensile strength, steels processed at sub-zero temperatures have better ductility (elongation).

4. Processing at sub-zero temperatures results in a greater spread between tensile strength and yield strength in the steels sub-zero processed.

5. A desired level of tensile strength is achieved by considerably smaller reduction (as in thickness, by rolling, or in wire, by drawing) at sub-zero temperatures than at room temperature.

6. Aging or "low-temperature" heat treatment enhances certain mechanical properties of rolled steels, especially if rolling has been done at sub-zero temperatures. The "aging" effect depends on the composition of the steel.

7. All steels, whether annealed or cold rolled (at room or at sub-zero temperatures) possess greater strength at sub-zero temperatures.

8. Cold-rolled steels, whether rolled at room or at sub-zero temperatures, have better ductility at sub-zero temperatures than at room temperature.

9. The distribution of ductility over 2-in. gage length is totally different at sub-zero temperatures than at room temperature.

10. This difference in distribution permits a more successful fabrication, by certain but not all forming processes, when such fabrication is carried out at lower than room temperature.

11. Cold-rolled stainless steels do not adversely suffer changes in their properties even after considerable time at temperatures up to 500 F. or even 800 F.

12. Tensile properties as well as ductility of cold-rolled stainless steels at temperatures up to and including 800 F. are lower than the same at room temperature.

Acknowledgments:

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DISCUSSION

MR. LEO SCHAPIRO.[1]—The properties that the authors have described, especially those from cold working at low temperatures, whether they are further improved by elevated temperatures or not, are very interesting, especially when it will be possible to have strength properties with much higher ductility values than available in present-day materials.

Mention has been made, both in the papers by Krivobok and Talbot and by Brace and Ziegler, in a brief way, about metals which are already processed. We in the aircraft industry are looking forward to the day when sheet metal will be processed in this fashion. We are interested because of the improved strength in forming, but we also need to know compressive strength properties, in addition to tensile strength properties. We expect that the compressive strengths will be lower by some low-temperature-rolling processes, and we hope that Mr. Krivobok and his colleagues will have such data for us by the time the materials are available commercially. We hope data will be available for material subsequently processed at high temperatures, also.

MR. R. H. CAUGHEY.[2]—There is no doubt that austenitic stainless steels with the developed level of mechanical properties described in this paper offers interesting possibilities to the aircraft designers and materials engineer. Two questions, however, seem pertinent at this time:

1. How will these materials treated in the manner described be expected to behave under high strain rates?

2. What is the relative effect of this treatment on the corrosion resistance of these materials?

These two questions are important in relation to the design of liquid-propellant rocket engines for aircraft in which white fuming nitric acid may be a component.

MR. L. L. WYMAN.[3]—Messrs. Krivobok and Talbot have quite justifiably emphasized one factor with which both of us had a considerable amount of experience during our association with the aircraft industry during the war. This is the manner in which the ductile properties of our metallic materials are measured—a subject on which, as many of you know, I have been harping since the war as an excellent example of simulated service testing.

What you find here is another outstanding example of where experimenters, having used these more logical methods of test, have come up with the real solution of a problem as well as an immediate application of a better test method.

In this application which is described to us, the test method is indeed a simulated service test. If Mr. Krivobok wants to bend this little gadget, as he calls it, the prime factor is not the prosaic "elongation in 2 in.," but rather the elongation of minute increments along the outer fibers right at the bend. When one begins to measure these elongations as he has done, the road is opened to the solution of the problem, and of times to considerable enlightenment concerning the large magnitude of local elongations as well.

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[2] Special Projects Dept., The M. W. Kellogg Co., Jersey City, N. J.

[3] Research Metallurgist, Knolls Atomic Power Lab., General Electric Co., Schenectady N. Y.

Mr. V. N. Krivobok (*author*).—Of the questions raised, the most important in our opinion is the effect of sub-zero.

We have not yet studied the corrosion resistance of these materials in specific corrosive media. It is our belief that working at sub-zero temperatures would have the same effect as working of these steels at normal temperatures. We do not expect this effect on corrosion resistance to be pronounced, at least in the majority of applications.

We wish to acknowledge that the photographs of the pieces bent at three different temperatures and shown in this paper have been most kindly loaned to us by our friend, Mr. A. E. Focke of the Diamond Chain Co.

In regard to the question on compressive properties, we worked with flat-rolled materials, which were of thicknesses not suitable for testing in compression. We are at the moment engaged in a very careful consideration of how compressive and torsional properties can be measured. We depend on our colleagues in aircraft and elsewhere to help us with this problem. As is well known from the work of the Bureau of Standards, it is not easy to measure compressive properties. But proper attention is being given to this problem.

Messrs. P. H. Brace[4] and N. A. Ziegler[5] (*by letter*).—Messrs. Krivobok and Talbot should be congratulated for their interesting presentation of the results of their monumental and precise piece of research. Their data surprisingly well confirm some of those obtained by the present discussors.[6]

Figure 6 is a vivid demonstration of one of the advantages justifying the application of sub-zero mechanical working—besides that of achieving super-high room temperature hardness and strength—not obtainable in this type of steels by conventional cold working; namely, that of achieving a certain value for the yield point and at the same time maintaining superior ductility. That fact is indeed of a major importance.

The other interesting fact pointed out by the authors is that "cold-rolled steels . . . have better ductility at sub-zero temperatures than at room temperature." This observation was demonstrated by Fig. 20 which showed several parts made from 18-8 steel, cold rolled at ordinary temperature and shaped by bending at ordinary and at several sub-zero temperatures. Although room-temperature bending caused cracks, none occurred when similar bends were made at sub-zero temperatures.

In connection with this observation, it might be of interest to refer to the remark by Sir Robert Hadfield that some of the austenitic chromium-nickel steels "could be cold-worked at -200 F., but not at room temperature."[7]

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[4] Consulting Metallurgist, Research Labs., Westinghouse Electric Corp., East Pittsburgh, Pa.

[5] Research Metallurgist, Crane Co., Chicago, Ill.

[6] N. A. Ziegler and P. H. Brace, "Hardening of Austenitic Stainless Steels by Mechanical Working at Sub-Zero Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 861 (1950).

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EFFECT OF RATE OF STRAIN ON THE FLOW STRESS OF GAS TURBINE ALLOYS AT 1200 AND 1500 F.*

By M. J. Manjoine[1]

Synopsis

The strength of metals and alloys and the factors which influence this strength are of paramount importance to the engineer who designs machines and to the metallurgist who develops new alloys and heat treatments for the parts of these machines. The results of tension tests at 1200 and 1500 F. of a group of high-temperature gas turbine alloys are reported. These tests include creep and creep-rupture tests, constant strain rate tests and high-speed impact tests. The strengths of these alloys are shown for a range of strain rates of 10^{-7} to 10^{+6} per hour. The factors which influence the flow-stress of a metal are discussed and a generalized flow-stress curve proposed. The short-time methods of comparing alloys and the limitations of these methods are reviewed.

In the gas turbine, stationary parts are combined with others that are rotating at extremely high speeds. In its operation, many of the parts are subjected to a range of loads and temperatures and some may even suffer impact. The machine designer, therefore, should know (for the material used) the rate of flow, the magnitude of the ductility, and the time to failure under a given stress and temperature, or the maximum stress and strain the material will withstand when deformed at various rates and temperatures. The metallurgist who is developing new alloys and heat treatments for these parts must, perforce, use quick methods of evaluating his progress. In addition, he should know how his evaluation compares with the relative strength under service conditions.

In this paper the ultimate strengths of several gas turbine alloys are given at 1200 and 1500 F. for a range of strain rates of 10^{-7} to 10^{+6} per hour. The factors which influence the strength of a metal or alloy are also reviewed.

Test Materials

Ten alloys were used in this investigation; four of these were wrought alloys and six were cast alloys. The compositions and heat treatment are given in Table I. In most cases, only the nominal compositions were available for the heats from which the specimens were prepared.[2] In the case of the wrought materials, the specimens were first, solution-treated; second, rough machined[3]; third, aged; and fourth, finished ground. The specimens of the cast alloys were tested as "precision cast"

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* Presented at the Fifty-third Annual Meeting of the Society, June 23–30, 1950.

[1] Westinghouse Electric Corp., Research Laboratories, East Pittsburgh, Pa.

[2] The specimens of these alloys were supplied by N. L. Mochel, Manager, Metallurgical Engineering, Westinghouse South Philadelphia Works.

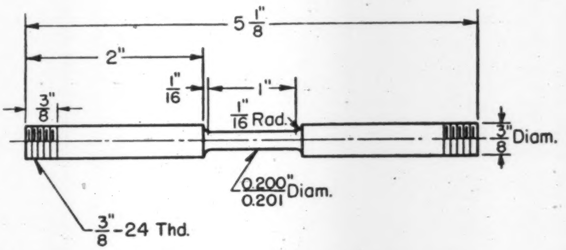
[3] Some materials were drop-forged to shape as indicated in table.

TABLE I.—COMPOSITION AND HEAT TREATMENT OF GAS TURBINE ALLOYS.

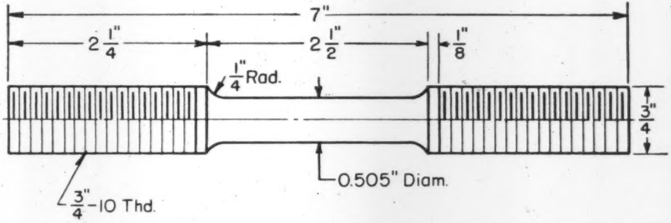
Material	Lot Number	Composition, per cent											Solution Treatment[g]	Aging Treatment[g]
		Carbon	Manganese	Silicon	Nickel	Chromium	Molyb-denum	Cobalt	Tungsten	Titanium	Iron	Others		
WROUGHT BAR														
K-42-B, Type II	1[a]	0.06[b]	...	...	41[b]	20[b]	...	24[b]	...	2.2[b]	balance	...	1790 F. 1 hr. O.Q.	1200 F. 90 hr. A.C.
	2	...	...	...	...	...	...	...	...	...	...	...	1750 F. 1 hr. W.Q.	1300 F. 24 hr. A.C.
	3	...	...	...	...	...	...	...	...	...	...	...	1950 F. 1 hr. W.Q.	1350 F. 20 hr. A.C.
Age Hardening Inconel	4	0.04	...	...	74[b]	14[b]	...	...	...	2.45[b]	...	Al 0.6[b]	1975 F. 6 hr. W.Q.	1300 F. 16 hr. A.C.
Nimonic 80	5	0.04	0.40	0.54	72.56	20.38	...	...	...	2.19	0.28	Al 1.24	1950 F.[f] W.Q.	1292 F. 16 hr. A.C.
Refractaloy	6	0.05	2.09	0.28	10.27	20.97	5.17	14.39	5.53	...	...	...	2350 F. 4 hr. O.Q.	1500 F. 240 hr. A.C.
	7[a]	0.11	1.97	0.25	20.1	20.2	8.3	30.1	3.8	...	15.2	...	2350 F. 4 hr. O.Q.	1500 F. 240 hr. A.C.
PRECISION CAST														
Stellite No. 21[c]	8	0.25[b]	...	...	...	28[b]	6[b]	65[b]	...	...	...	...	None	None
	9	...	...	...	...	...	...	...	...	...	...	...		
Stellite No. 23	10	0.4[b]	...	...	...	28[b]	...	balance	5.6[b]	...	...	...		
	11	...	...	...	...	...	...	...	...	...	...	...		
	12	...	...	...	...	...	...	...	...	...	...	...		
Stellite No. 27	13	0.4[b]	...	...	½ balance	28[b]	6[b]	½ balance	...	...	...	...		
	14	...	...	...	...	...	...	...	...	...	...	...		
Stellite No. 30[d]	15	0.4[b]	...	...	16[b]	25[b]	6[b]	balance	...	...	2 max.	...		
	16	...	...	...	...	...	...	...	...	...	...	...		
Stellite No. 31[e]	17	0.4[b]	...	...	10[b]	25[b]	...	balance	7[b]	...	...	...		
	18	...	...	...	...	...	...	...	...	...	...	...		
Stellite No. 6	19	...	...	...	...	33[b]	...	55[b]	6[b]	...	...	7.5 max.[b]		

[a] Drop forged bars.
[b] Nominal value.
[c] Vitallium.
[d] 422-19.
[e] X-40.
[f] Probable solution temperature.
[g] O.Q. = oil quench.
A.C. = air cool.
W.Q. = water quench.

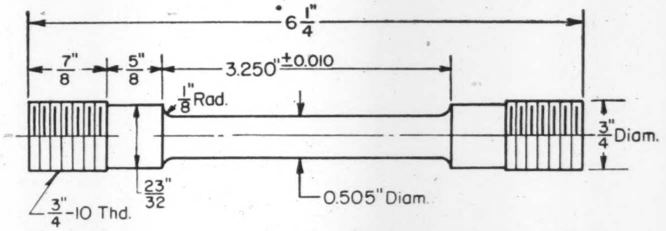
to shape by Haynes Stellite Co. The shapes of the specimens used are given in Fig. 1. The specimen in Fig. 1(*a*) was used for the constant strain rate and high-speed or tension impact tests of all the alloys. The creep and creep-rupture tests of the cast alloys were made on specimens precision cast as in Fig. 1(*b*). The specimen shown in Fig. 1(*c*) was for those of the wrought alloys.


5 1/8"
2"
1/16"
1"
3/8"
1/16" Rad.
3/8" Diam.
0.200"/0.201 Diam.
3/8"-24 Thd.

(*a*) Constant Strain Rate and High-Speed Test Specimen.


7"
2 1/4"
2 1/2"
1/8"
1/4" Rad.
3/4"
0.505" Diam.
3/4"-10 Thd.

(*b*) Creep and Creep-Rupture Test Specimen—Precision Cast Alloys.


6 1/4"
7/8"
5/8"
3.250"±0.010
1/8" Rad.
3/4" Diam.
23/32"
0.505" Diam.
3/4"-10 Thd.

(*c*) Creep and Creep-Rupture Test Specimen—Wrought Alloys.

FIG. 1.—Test Specimens.

TESTING MACHINES

To cover the range of strain rates for the tests reported herein, four types of testing machines were employed.

High-Speed Tension Machine:

For the extremely rapid tests at a rate of strain of 7.2×10^5 per hour the high-speed tension machine (**1**)[4] was used. This machine employs a flywheel to store the energy necessary to pull a specimen to rupture in about one millisecond. The load and elongation of the specimen are recorded on an oscillogram. A photograph, Fig. 2, shows this machine and its associated apparatus.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 947.

Constant Strain Rate Machine:

The intermediate range of strain rates from 10^{-3} to $2 \times 10^{+3}$ per hour are obtained with the constant strain-rate tension machine. This screw driven tension machine (2) gives an autographic record of load *versus* elongation. Changes in strain rate are accomplished by interchanging and removing speed reducers in the drive system.

Creep and Creep-Rupture Machines:

For the lower rates of strain the tests were made at constant load on the screw-driven creep-rupture and lever-arm creep machine (3). A sketch of the installation of these machines at the Westinghouse Research Laboratories is given in Fig. 3. In the screw-driven creep-rupture machine (left, Fig. 3) the test specimen is loaded by a screw jack through a stiff spring in series with the specimen. The load is maintained by keeping a constant deflection of the spring. A continuous elongation-time curve up to rupture is automatically recorded without the use of an extensometer on the test specimen. The machine has a capacity of ten tons. These units are used for the creep-rupture tests at the higher rates of strain from 10^{-4} to 10^{-2} per hour.

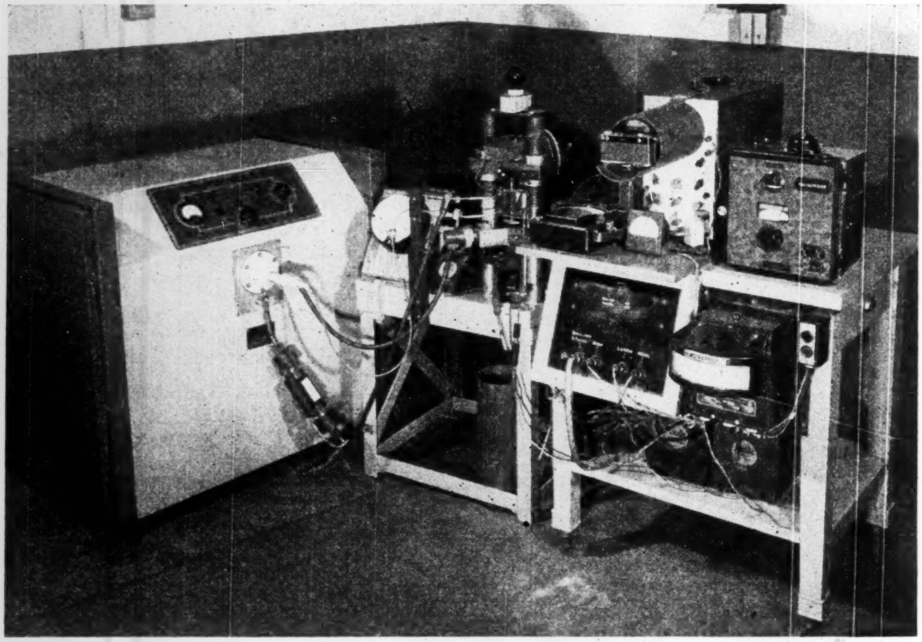

FIG. 2.—High-Speed Tension Machine and Associated Apparatus.

The creep tests and the longer creep-rupture tests were made in the lever-arm creep machines (right, Fig. 3). These machines[5] are conventional in the manner of load application and have a capacity of six tons. The extensometer gives a continuous indication of the elongation up to rupture; its least count is $8\frac{1}{3}$ microinches per inch for the standard specimen, Fig. 1(*c*).

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[5] Built by Baldwin Locomotive Works.

TEST RESULTS

For the constant strain rate tests, ultimate stress and strain at rupture are plotted in Figs. 4 and 5. These data were taken from the autographic record of load and elongation obtained on the constant strain rate and high-speed machines. For the creep and creep-rupture tests, the following data (Table II) are given where possible: alloy name and number, test temperature, strain intercept,[6] minimum creep rate, strain and time at the transition point,[7] strain and time at rupture, original and final Vickers hardness (DPH)[8] and reduction of area. A plus sign following the datum indicates that the test was interrupted before the "point," under which the datum is listed, had been reached. Any exceptions in the data are listed in the footnotes.

Flow Stress versus Strain Rate:

The "flow" or "yield" stress of a material is that stress under which the material will deform plastically. A particular "flow-stress," therefore, must be further qualified. In the data reported here, the "flow-stress" will refer to the stress at the maximum load for any of the various types of tests. For the tests at constant load, this flow-stress is constant throughout the test, the strain rate used in plotting these data is the minimum creep rate. In Fig. 4 this flow stress is plotted for the alloys tested at 1200 F. as a function of the rate of strain. From the creep tests to the high-speed tests, the rate varies from 10^{-7} to 10^{6} per hour. To cover this range of rates it was necessary in most cases to use specimens from different heats of an alloy but with the same nominal composition. The test points for the various alloys and the different heats

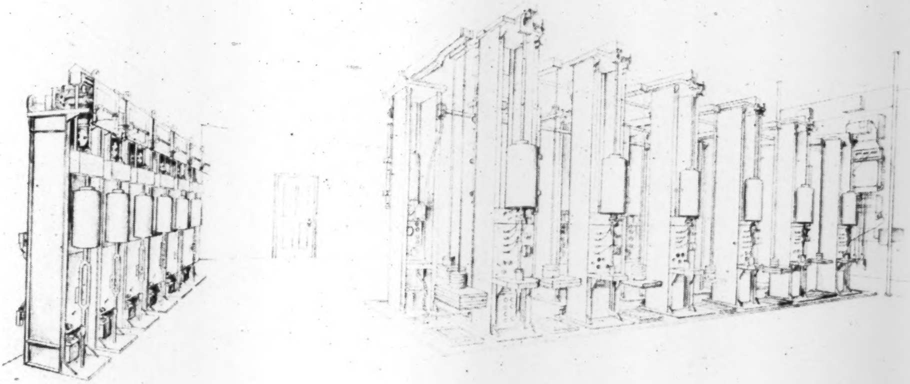

FIG. 3.—Group Installation of Creep and Creep-Rupture Machines.

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[6] Intercept on strain axis at zero time of tangent to creep curve at minimum rate.

[7] Transition from second to third stages of creep where creep rate has increased 10 per cent over the minimum rate.

[8] At a point not subjected to stress.

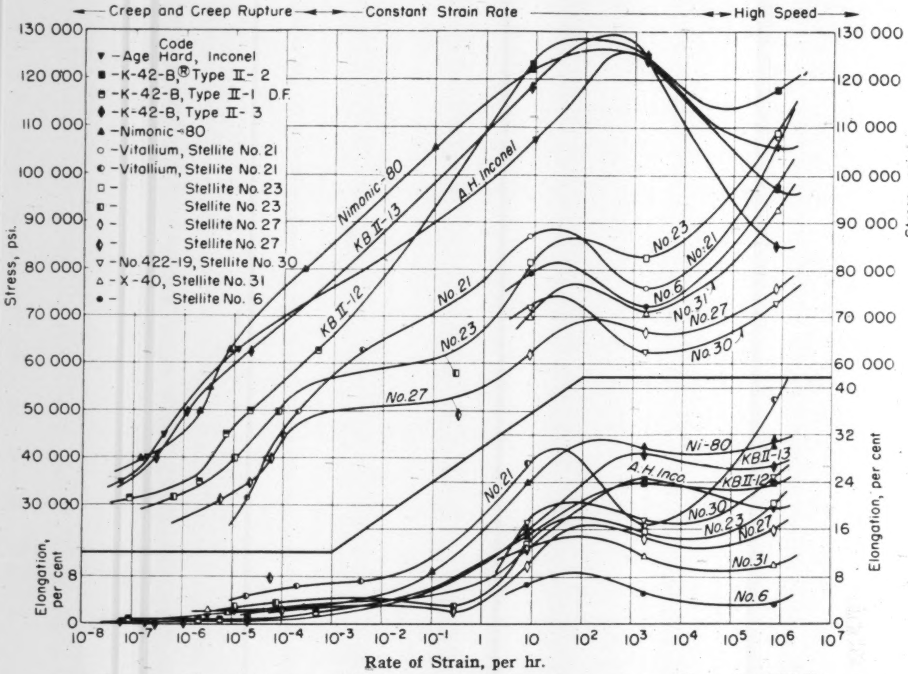


FIG. 4.—Flow Stress *versus* Strain Rate of High-Temperature Alloys at 1200 F.

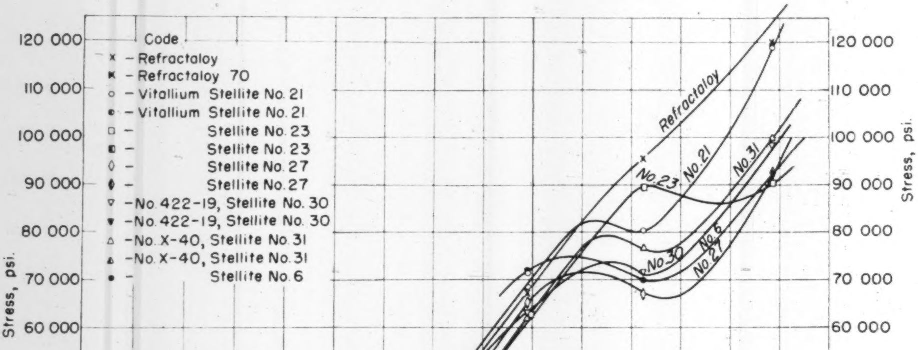


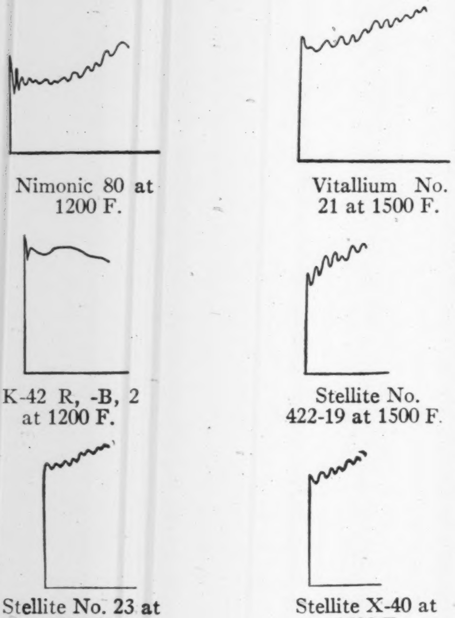
FIG. 5.—Flow Stress *versus* Strain Rate of High-Temperature Alloys at 1500 F.

TABLE II.—CREEP AND CREEP-RUPTURE TEST DATA.

Material	Temperature, deg. Fahr.	Stress, psi.	Intercept, per cent[a]	Creep Rate, per hr.	Transition Point[b]		Rupture		Diamond Pyramid Hardness 100-kg., No Stress		Reduction of Area, per cent
					Time, hr.	Strain, per cent[a]	Time, hr.	Strain, per cent[a]	Original	Final	
K-42-B, Type II, 1	1200	63 000	0.70	4.6×10^{-4}	22	1.70	25.3	2.1	316	324	3.0
		50 000	0.31	2.1×10^{-5}	280	0.93	311	1.12	330	340	2.4
		45 000	0.07	6.8×10^{-6}	570	0.46	680	0.73	323	307	1.4
		35 000	0.15	2.1×10^{-6}	825	0.36	1192	0.7	319	300	...
		31 500	0.16	9.0×10^{-7}	1800	0.39	2863	0.85	347	306	1.2
K-42-B, Type II, 3	1200	63 000	0.30	2.2×10^{-5}	...	...	77	0.47	301	316	7
		50 000	0.21	1.2×10^{-6}	...	...	204	0.30	297	342	6
		40 000	0.20	3.0×10^{-7}	...	...	781	0.31	298	318	1.4
Age Hardening Inconel, 4	1200	63 000	0.4	1.2×10^{-5}	88	0.51	89	2.3	...	308	5
		55 000	...	...	...	...	310	1.23	...	328	5
		50 000	...	...	...	...	650	1.20	...	350	1
		45 000	0.26	4.3×10^{-7}	910	0.30	915	0.30	284	340	...
		40 000	...	1.5×10^{-7}	...	...	540	[e]	286	329	...
		40 000	0.24	2.6×10^{-7}	...	...	1168	0.27	301	353	1.8
		35 000	0.145	6.0×10^{-8}	1340[c]	0.16	1455[c]	0.24	298	336	...
Nimonic 80, 5	1200	80 000	0.60	2.6×10^{-4}	25	1.33	59	2.68	281	286	8.7
		63 000	0.45	8.3×10^{-6}	100	0.54	460	2.13	282	297	7.9
		55 000	0.41	3.5×10^{-6}	1000	0.72	2098	2.3	283	329	3.8
		50 000	0.24	2.2×10^{-6}	1490+[d]	0.60+	...	...	272	326	...
		40 000	0.19	1.5×10^{-7}	2110+	0.22+	...	...	282	320	...
Refractaloy 70, 7	1500	25 000	0.25	3.9×10^{-3}	4.3+	2+[f]	...	...	366	341[f]	27.2[g]
		20 000	0.33	8.9×10^{-4}	10	1.3	52	20.5	374	...	...
		18 000	0.30	2×10^{-4}	35	1.1	120	15.8	426	392	25
		16 000	0.25	2.1×10^{-5}	160	0.62	447	9.68	411	385	17.7
		14 000	0.24	$<1.7 \times 10^{-6}$	330+	0.3+[f]	...	...	434	395[f]	12.3[g]
		12 000	0.43	1×10^{-7}	4500	0.55	6015+	0.68+	443	367	...
Vitallium No. 21, 8	1200	63 000	6.3	3.7×10^{-3}	3	7.2	3.1	7.3	320	334	14.1
		50 000	2.45	2.0×10^{-4}	50	3.0	68	6.5	317	318	12.2
		40 000	0.72	4.7×10^{-5}	145	1.37	183+	1.7+	336	402	2.3+
		31 500	0.21	2.0×10^{-5}	250	0.77	1140	4.47	320	443	8.7
		31 500	0.16	2.0×10^{-5}	1800	4.25	1960	4.83	315	438	...
	1500	20 000	0.92	1.1×10^{-3}	30	4.35	41	8.02	305	368	19.8
		16 000	1.46	1.8×10^{-4}	77	2.96	104	5.65	312	414	8.6
		10 000	0.47	4.1×10^{-5}	442	2.33	668	6.7	308	502	16.1
		8 000	0.40	7.0×10^{-6}	1600	1.50	2085	2.88	336	440	...
Stellite No. 23, 10	1200	50 000	1.77	8.0×10^{-5}	157	3.05	186	3.66	336	351	8.3
		40 000	0.86	1.1×10^{-5}	1150	2.07	1475	2.76	350	434	4.6
		31 500	0.28	7×10^{-7}	1600+	0.35+	...	...	347	462	0.5
Stellite No. 23, 12	1500	30 000	0.90	3.3×10^{-3}	12.7	...	13.3	6.23	328	400	10.2
		25 000	0.60	8.3×10^{-4}	21+[h]	2.34+	...	...	337	454	3
		24 000	2.16	1.5×10^{-4}	104	3.7	111	4.10	316	540	6.4
		20 000	1.01	1.1×10^{-4}	211	3.5	216	4.21	340	500	10.6
		16 000	0.80	5.9×10^{-6}	1160	1.5	1194	1.93	320	464	6.7
		12 000	0.49	8×10^{-7}	1821+	0.63+	...	...	331	540	...
Stellite No. 27, 13	1200	45 000	1.35	1.0×10^{-4}	...	...	125	2.67	245	318	3.8
		40 000	2.1	5.1×10^{-5}	790	6.2	940	8.08	242	319	13.4
		35 000	0.15	2.4×10^{-5}	1000+	2.46+	...	...	247	307	...
		31 500	0.60	5.8×10^{-6}	1144+	1.27+	...	...	253	336	...
	1500	25 000	1.95	6.4×10^{-3}	11	8.91	22	20.08	256	...	24.1
		20 000	1.72	7.5×10^{-5}	284	4.0	322	6.95	263	326	12.2
		16 000	1.23	3.4×10^{-6}	1600	1.8	2118+	2.23+	255	319	...
		12 500	0.78	5.0×10^{-7}	2877+	0.93+	...	...	258	...	1.6+
Stellite No. 30, 16	1500	30 000	1.02	1.3×10^{-3}	15	3.0	17.5	4.09	302	419	5.3
		27 500	1.36	2.0×10^{-3}	20	5.45	26	7.77	283	399	...
		27 500	2.06	1.6×10^{-3}	95	3.63	115	4.54	315	400	...
		25 000	1.36	6.5×10^{-5}	205	2.76	240	3.74	320	413	3.1
		20 000	1.13	1.1×10^{-5}	1010	2.31	1056	3.19	277	422	3.1
		17 500	0.72	3.6×10^{-6}	2000	1.45	2149	1.88	304	422	...
		16 000	0.75	1.5×10^{-6}	2110+	1.06+	...	...	312	406	1.6
Stellite No. 31, 18	1500	35 000	1.20	7.4×10^{-3}	9.5	9.45	15	16.75	281	446	14.7
		30 000	2.71	3.8×10^{-4}	117	7.35	135	9.8	280	416	12.8
		26 000	3.45	8.2×10^{-5}	240	5.5	325	8.77	277	426	16.5
		16 000	1.13	6.7×10^{-7}	2489+	1.30+	...	...	307	412	3.3
		14 000	0.88	4.0×10^{-7}	1987+	0.96+	...	...	287	439	...

[a] Total Strain.
[b] Transition from second to third stage of creep where creep rate has increased 10 per cent over the minimum rate.
[c] Broke at fillet.
[d] Plus sign indicates that test was interrupted before this point was reached.
[e] Broke in threads.
[f] Specimen overheated.
[g] Specimen broken during overheating.
[h] Pulled out of threads—test discontinued.

are coded to facilitate the following of these curves. Also plotted in the lower part of Fig. 4 is the elongation at rupture. The same data for tests at 1500 F. are given in Fig. 5. A thin line is used to separate the "elongation" from the "stress" curves. The elongation curve for Refractaloy[9] alloy at the low rates of strain crosses this line; the curve is marked by two vertical dashes to distinguish it from the other curves.



Nimonic 80 at 1200 F.

Vitallium No. 21 at 1500 F.

K-42 R, -B, 2 at 1200 F.

Stellite No. 422-19 at 1500 F.

Stellite No. 23 at 1200 F.

Stellite X-40 at 1500 F.

Fig. 6.—Original Oscillograms of Some High Speed Load-Elongation Curves. Strain Rate = 200 per sec. or 7.2×10^5 per hr.

DISCUSSION OF RESULTS

In general, the strain at the ultimate stress is practically the same as that at rupture. An alloy, therefore, strain hardens and stretches uniformly throughout the test. To illustrate the shape of the load-deflection curve at high rates of strain, some tracings of original oscillograms are reproduced in Fig. 6. At 1200 F. the diagrams for Stellite No. 23 alloy and Nimonic 80 alloy show that the load increases from the yield strength to fracture. The diagram for Vitallium No. 21 alloy illustrates the large elongation of this alloy at the high rate of strain. The vibrations superimposed on the diagrams are the natural frequency vibrations (longitudinal and lateral) of the load recording system. The load on the specimen is given by a smooth curve drawn through the average of these vibrations.

Flow Stress versus Strain Rate:

Since the load and strain increase up to rupture, the true stress increases also. However, in Figs. 4 and 5 only the "stress" based on the original area is given. Refractaloy, 6 alloy is one of the exceptions, since the strain at the ultimate is usually less than one-half the rupture strain, this alloy is remarkable in that the increase in strength with strain rate is very large.

The ultimate strength of each of the wrought alloys in Fig. 4 varies in a similar manner with strain rate; nevertheless the curves for these alloys cross several times during this range of rates of about 10^{-7} to 10^6 per hour. The relative superiority of any of these alloys, therefore, depends on the particular rate at which they are compared. The strength of these alloys, in general, increases with rate of strain, reaches a peak at a strain rate of about 10^2 to 10^3 per hour, and then drops off with further increase in rate for several "cycles."[10] The curves for the cast alloys have the same general "shape." In general, the cast alloys have lower strength than the wrought alloy at 1200 F. At the highest rate, several of the cast alloys are stronger than some of the wrought alloys.

At 1500 F., Fig. 5, only one wrought

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[9] Trade Mark Reg. U. S. Pat. Off.

[10] Strain rate is plotted logarithmically (base 10). Each "cycle" is a 10 fold change.

alloy, Refractaloy, was tested. At a rate of strain of 10 per hour, the strengths of all the alloys fall within a band of 10,000 psi. At lower or higher strain rates the curves diverge. As was found at 1200 F., the strength curves at 1500 F., although similar in shape, cross each other, and the relative superiority varies with the strain rate at which they are compared.

Elongation versus Strain Rate:

The performance of these gas turbine alloys is often limited by their ductility. The elongation in 1 in.[11] expressed in per cent plastic strain is plotted at the bottom of Figs. 4 and 5. At 1200 F., bottom Fig. 4, the general pattern of the elongation curves is similar to that of the strength curves. In most cases, the elongation increases with strain rate up to about a rate of 10 per hour and then reaches a peak or levels off. Some of the cast alloys again begin to increase at the extremely high rates. In general, these alloys have good ductility at the higher rates of strain, although they may be rather brittle when deformed at the low rates of strain. At the bottom of Fig. 5, the elongations of these alloys at 1500 F. are given. Although Stellite No. 21 has next to the lowest ductility at the low rates of strain, it has the greatest at the high rates. Refractaloy, the only wrought alloy, shows an opposite effect.

The elongation values are useful in predicting the rate to work a metal at a given temperature and in preventing the formation of cracks. The force necessary to work the metal is also of prime importance. The most desirable rate to work the metal at a given temperature will be a compromise and will probably be that rate near the peak of the ductility curve and on the side of the peak where the strength is lower.

Test Data:

Each datum point in Figs. 4 and 5 represents the result of only a single test with the exception of several check tests made to investigate possible scatter. In the check tests on Nimonic 80 and Refractaloy alloys at the high-speed strain rate of 7.2×10^5 per hour the elongations checked within 5 per cent. Check tests of Stellite No. 21 at a rate of 8 per hour gave a two to one change; in this case, however, it was found that failure of the specimen with the lower ductility occurred in a large grain near the fillet of the specimen. In these check tests the ultimate stress varied less than 8 per cent for most of these alloys. All of the specimens did not come from a single heat but from heats of the same nominal composition. It is recognized that especially in these cast alloys the properties of one heat may be different from another because of the influence of pouring and mold temperatures as well as small composition changes. It was felt that these variations would not materially affect the general shape of these curves because of the large over-all change in stress which occurs over the range of strain rates.

The initial heating period is an important aging period for the test specimens (especially for those of the cast alloys which are tested "as cast"). In the creep or creep-rupture tests the specimen is heated about 16 hr. before the load is applied. In the constant strain rate test the heating time is about 3 hr. and in a high-speed test it is only 1 or 2 min. For the tests at 1200 F., it can be shown (**4, 5**) that the preheating without stress for a period up to 16 hr. will probably have very little effect on the hardness and, presumably, on the ultimate strength. At 1500 F., aging takes place more rapidly; the results of the creep and creep-rupture tests, therefore, are of specimens in a slightly different metallurgical state.

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[11] For the creep tests the elongation is measured over a longer gage length. The gage length for the cast creep specimen is 2-in. and that for the wrought specimen is 3-in. However, since very little necking was observed this difference is gage length does not cause a serious change in elongation values.

TABLE III.—COMPARISON OF ENDURANCE AND ULTIMATE STRENGTHS AT 1200 AND 1500 F.

Materia		Temperature, deg. Fahr.	Endurance Strength 10^8 Cycles, psi.[a]	Ultimate Strength at Rate of 10^3 per hr., psi.	Endurance Ratio
K-42-B-13	3	1200	63 000	128 000	0.492
K-42-B-12	2[a]	1200	58 000		0.459
		1200		126 500	
A.H.Inconel	4	1200	58 000	125 500	0.462
X-40, Stellite No. 31	17[a]	1200	56 000		0.779
				72 000	
Nimonic 80	5	1200	55 000	125 500	0.438
Stellite No. 23	10	1200	44 000		0.527
	11			83 500	
Vitallium No. 21	8	1200	44 000		0.571
	9			77 000	
Stellite No. 27	13	1200	41 000		0.602
	14			68 000	
Stellite No. 23	10	1500	38 000		0.431
	11	1500		88 000	
X-40, Stellite No. 31	17[a]	1500	34 000		0.436
		1500		78 000	
Stellite No. 21	8	1500	33 000		0.413
	9	1500		80 000	
Stellite No. 27	13	1500	32 000		0.464
	14	1500		69 000	

[a] Different heat of alloy, described in Reference (6).

Methods of Comparing Alloys:

In the sorting or comparison of alloys for creep, creep-rupture, or other types of service, the usual procedure is to make short-time tension, constant strain rate, or hot hardness tests to determine the relative properties. All three of these comparison methods involve plastic flow of the alloys at a fairly fast rate of strain and some measure of the alloy's resistance to this deformation. The strain rate commonly used in a short-time tension test or a comparable constant strain-rate test is about 2 per hour. The curves in Figs. 4 and 5 show the possible errors in sorting alloys by these methods. If the flow stresses at 1200 F. and a rate of 2 per hour are compared with those at a creep rate of 10^{-5} per hour, it can be seen in Fig. 4 that the wrought alloys are stronger at both rates than the cast alloys. If, however, the cast alloys are compared, then the strongest alloy at a rate of 2 per hour, Stellite No. 21, is the weakest at a rate of 10^{-5} per hour. Also for the wrought alloys, K-42-B type II alloy with the No. 2 heat treatment is next to the strongest at the high rate but the weakest at the low rate.

At 1500 F., Fig. 5, the strength of any of the alloys at a rate of 2 per hour is within 16 per cent of any other; but again the order of the strength of these alloys is, in most cases, reversed when compared to that at the low rates. This fact is brought out to caution those who use testing techniques involving a particular rate of strain to determine the relative merits of alloys which are to be used at a greatly different rate of strain. For example, the rate of loading in a fatigue test is very high, the strength of a material at a comparable rate of loading or strain should be a reasonable measure of the relative endurance strength. The endurance strengths (6) of some of the alloys reported herein are given in Table III. A comparison is made between the endurance strength at 10^8 cycles and the ultimate strength at a strain rate of 10^3 per hour. The "Endurance Ratio" at 1200 F. is about 0.44 to 0.49 for the wrought alloys and about 0.53 to 0.60 for the cast alloys with the exception of alloy X-40. At 1500 F., the four cast alloys are within

6 per cent of the endurance ratio of 0.44 in spite of the fact that the two types of tests were made on different heats of the alloys.

FACTORS INFLUENCING THE FLOW STRESS OF A METAL

In the light of the data presented here, it is reasonable to review the factors which influence the flow stress of a metal. These factors are: (*1*) strain, (*2*) rate of strain, rate of loading, and time to failure, (*3*) temperature, (*4*) metallurgical state, and (*5*) state of stress.

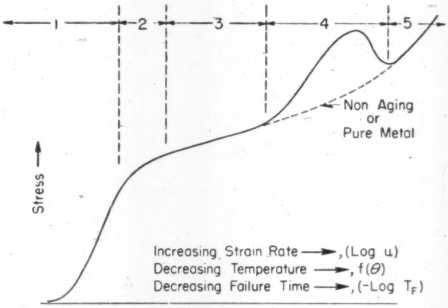

FIG. 7.—Characteristic Flow-Stress Curve for Metals.

Because of the interrelation of these factors, it is possible to express their effect qualitatively on the flow stress of a metal. The characteristic flow-stress curve for metal as a function of rate of strain, time to failure, or temperature is presented in Fig. 7. If a metal of a particular metallurgical state is deformed in a given manner at a constant temperature, the stress, with which it will resist deformation to a given strain, as a function of the log rate-of-strain or of log time-to-failure is given by this flow-stress curve.

At the extremely high rates or short times at stress, zone 5, the stress increases nearly as a power function of the log of strain-rate or minus log of time-to-failure. In zone 4 the rate of strain is lower and the time to reach a given strain is longer; thus metals which "age" due to the diffusion of relatively fast atoms (such as oxygen, carbon, and nitrogen) (**7**) will show an increase in strength as the strain rate is decreased until the "aging" is complete. As the strain rate is further decreased, overaging occurs and the strength decreases. Metals which do not exhibit this "aging" follow the dashed curve in zone 4. In zone 3 no anomalies occur and the stress is practically linear with respect to the log of creep-rate or of time-to-failure. At lower rates of strain, zone 2, metallurgical changes take place in the metal, and accompanying this long-time instability there is a loss in strength when the rate is decreased. The mechanism of flow may also change in this zone as well as the relative strength of the grain and grain boundaries. In zone 1 at the extremely low rates of strain the stress decreases further with decreasing rate of strain. For many materials, the stress in this zone can be expressed as a hyperbolic function of the rate of strain (**8**).

The characteristic curve in Fig. 7 is therefore a qualitative representation of the flow stress as a function of rate of strain, time-to-failure, or temperature since the exact functions relating these variables in all cases are not known (**9**). However, much work (**10, 11, 12, 13, 14**) has been done and fairly accurate quantitative results can be obtained for some materials over a limited range of the variables.

Strain:

The flow-stress has been defined as that stress at which a material will deform plastically. Since strain hardening may occur, it is necessary further to qualify the flow-stress by giving either the amount of plastic strain to be associated with it or some other criterion, such as, "at the ultimate" or "at the minimum creep rate." For example, the flow stress at 0.2 per cent plastic strain is equivalent to the yield strength at 0.2 per cent offset. Strain will here refer to the deformation which occurs subsequent to the initial treatment of a metal or alloy; that is, after prior processing and heat treatment. Thus the characteristic flow-stress curve for a metal to be complete should be a family of curves, one for each value of plastic strain to be considered. This family of curves converges to a narrow band for the lower rates or for high temperatures (**15, 16**).

Rate-of-Strain, Rate-of-Loading, and Time-to-Failure:

These three factors are considered together since they are all related to time. Rate of strain is the time derivative of plastic strain or, more popularly, the speed of deformation.

The flow stress at the maximum load as a function of the log of the rate of strain for four metals or alloys is plotted in Fig. 8 to illustrate how these metals conform to the characteristic curve[12]

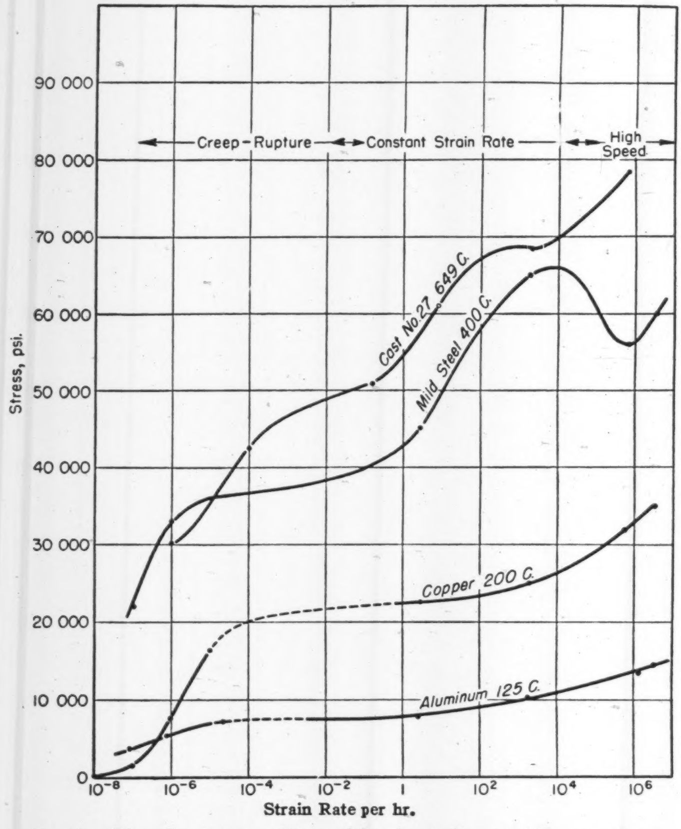

FIG. 8.—Flow Stress *versus* Rate of Strain at Constant Temperature.

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[12] The characteristic curve also qualitatively represents the internal friction *versus* temperature (**17**).

of Fig. 7. The data[13] are taken from the types of test shown at the top of the curve sheet. In all cases the material used in the creep tests is of the same nominal composition but of a different heat than the corresponding material used for the other strain rates. Two of the metals are "non-aging" materials, and the other two undergo the short-time instability described for zone 4, Fig. 7. All four materials exhibit a long-time instability which gives the "knee" of zone 2, Fig. 7.

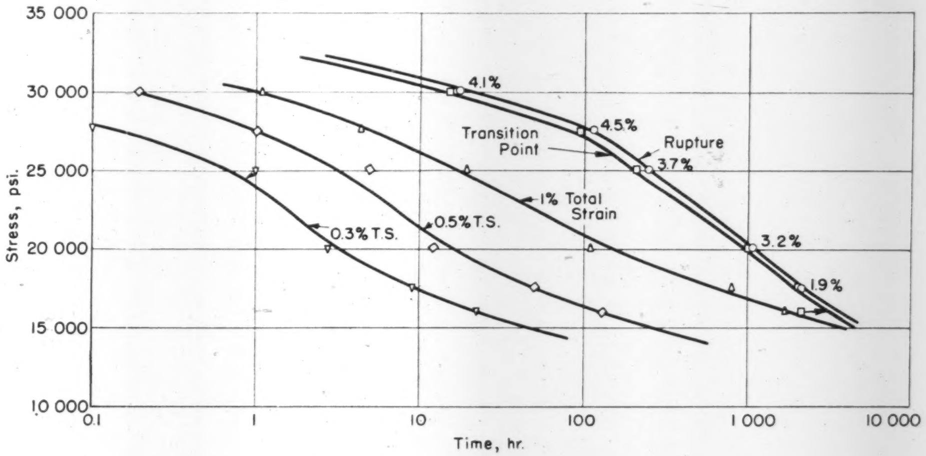

FIG. 9.—Design Curves. Cast Stellite 30 at 1500 F.

Rate of loading is proportional to the time derivative of elastic strain. The flow-stress at the proportional limit is a function of the rate of loading (20).

The mechanism of flow and failure becomes more complex when the load and, therefore, the stress are varied with time. If the load varies slowly with time, it is possible to determine the deformation or failure time by integrating the instantaneous effect. However, in many machines the parts are subjected to a rapidly alternating load, and this loading may be combined with a steady load to produce a pulsating load. In either case the influence of the three factors—rate of strain, rate of loading, and time (at load)—must be considered to obtain a clearer picture of the mechanism of flow and failure (21).

To discuss the other factor "time-to-failure," it will be necessary to define "failure." In many machines or structural parts, failure is considered to have occurred if the material exhibits any plastic flow; for these parts the stress at the proportional limit is the stress at failure. In other parts a certain deformation can be allowed; failure in these cases occurs when this strain is reached. If a considerable strain can be tolerated, then the material can be used to the transition point or to rupture. The flow-stress curves as a function of time for each of these criteria of failure form a family of curves for a given alloy. These "failure" curves are commonly called "Design Curves" and are illustrated in Fig. 9. The rupture strain is tabulated adjacent to the rupture point to illustrate the manner in which the strain decreased for the longer rupture times.

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[13] The source of the data is as follows: Cast Stellite No. 27 at 1200 F., this paper; constant strain rate tests of mild steel at 400 C., copper at 200 C., and aluminum 125 C. from reference (**2**); creep tests for mild steel from creep data (**18**); creep test of copper by E. A. Davis (**19**); and creep tests of aluminum from unpublished work at Westinghouse Research Labs., by E. A. Davis.

It can be seen that Fig. 9 is similar to zones 1, 2, and 3 of Fig. 7 if minus log of time is substituted for log of time so that time decreases to the right.

Temperature:

Much of the discussion above refers to the flow of metals at only one constant temperature, although a brief reference has been made to the interrelation of the factors influencing the flow of metals such as by the use of the chemical rate theory. If a metal of a particular metallurgical state is deformed at a constant rate of strain, the flow-stress, at a given strain, with which the metal will resist the deformation—as a function of temperature—can be represented by the characteristic curve, Fig. 7, in which the temperature decreases to the right. It is to be understood, however, that the deformation occurs at a constant temperature for each "point" on the curve. Starting at a low temperature, zone 5, the stress decreases at a higher temperature (toward the left). If the metal "ages" at the higher temperature, the strength increases, zone 4. This zone is called the "blue brittleness" range for carbon steels. In general, it represents a low-temperature instability of the metal.

Again at higher temperature, zone 3, the stress continues to decrease. At a still higher temperature, zone 2, or zone of high-temperature instability, metallurgical changes begin to take place or are completed. This includes such changes as recovery, recrystallization, phase changes and precipitation. For the higher temperatures of zone 1 and if no further metallurgical changes take place the stress will gradually approach zero at the melting point of the metal.

The absolute temperature of these zones can be shifted by the other factors. For example, the "blue brittleness" range can be shifted to a higher temperature by increasing the rate of strain (**16**). A metallurgical change will take place, therefore, over a range of temperatures or over a range of times at a constant temperature. The knowledge of the strength of an alloy as a function of rate of strain, alone, is not usually sufficient information to determine the suitability for a given service and service life but can be used to supplement the data of the design curves, Fig. 9, and to aid in their extrapolation.

Metallurgical State:

The following are the factors which influence the metallurgical state.

Composition.—The strength of the base metal can be increased by alloying. Some alloying constituents improve the strength of the grain boundaries or that of the grains while others improve that of both (**22, 23**). The susceptibility of the alloy to heat treatment is greatly determined by the hardener content (**16**). The recovery, recrystallization and aging temperatures depend largely on the composition, as do strain hardening (**24**), the capacity for cold work, and the grain size.

Prior processing.—For the gas turbine alloys, the influence of prior processing (**22, 25, 26, 27**) is at present under considerable study, although its effect in many alloys can be diminished by suitable heat treatment (**5**).

Heat treatment.—For an alloy of a given composition, the size of the structural constituents or grain size can be varied by heat treatment to increase the strength of a metal. However, the relative strength of the grain and grain boundaries for the given service conditions will dictate whether the grain size should be increased or decreased.

The strengths of the grains and grain

boundaries are each influenced by the same factors which have been discussed above for the flow-stress, and, therefore, the characteristic curve will probably describe the variation of their strengths. The relative strength under given service conditions for a limited range is shown in Fig. 10. The strengths of the grains and grain boundaries as functions of temperature, time or rate[14] are given by the solid curves and their combined strength by the dashed curve. In the characteristic curve their combined strength contributes to the change shown in zone 2. The point where the two strengths are equal is called the "equicohesive strength." The break in the curve of rupture strength as a function of time has been attributed to this intersection of the strengths (**28**). Since the strengths of the two constituents are affected differently by the various factors, their intersection can be shifted over a wide range of temperature, time, and rate.

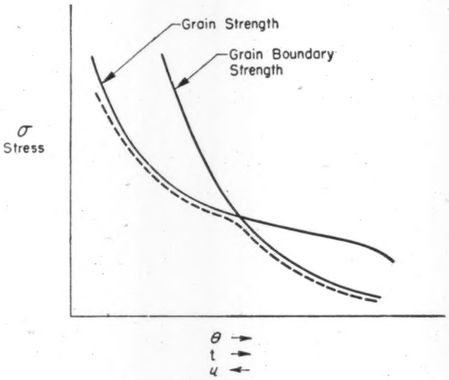

FIG. 10.—Equicohesive Strength.

Alloys which can be hardened by heat treatment are superior for many applications because the hardness is usually more homogeneous. The characteristic-curve anomalies in zones 2 and 4 have been attributed to metallurgical changes. The changes in zone 4 were termed "short-time" ones, since they are caused by elements which diffuse rapidly, while those in zone 2 were called "long-time" ones. This is used in a relative sense since at low temperatures the changes in zone 4 may take considerable time. When an alloy is prepared or heat treated in such a manner that aging, precipitation hardening, or a phase change occurs then it is obvious that the aging change in zone 4 (**29**) and the precipitation or phase change in zone 2 may be considerably reduced or entirely eliminated. An example of this can be seen in Fig. 4 for the wrought alloys which were heat treated by precipitation hardening. Aging effects are usually reduced by increasing the aging temperature. A comparison of the cast alloys in Fig. 4 (1200 F.) and Fig. 5 (1500 F.) shows that the changes at both the high and low rates of strains have been reduced and that the knee or peak of the zones has been shifted to a higher rate of strain with increasing temperature (**16**). Attention is also called to the large difference in the strength of K-42-B alloy in the creep-rupture range for the two heat treatments.

For the cast cobalt-base alloys reported here, a phase change occurs when the alloys are aged (**27**). This phase change and associated metallurgical changes account for the anomalies shown in Figs. 4 and 5. Heat treatment can be expected to change the shape of the family of curves for various strains, since aging has a large effect on the yield strength of these alloys when tested below the aging temperature and only a small effect on the ultimate strength and the creep strength (**4**). The strength curves cannot be extrapolated, therefore, unless the nature of these metallurgical changes is known.

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[14] Note that the abscissa values are reversed from that of Fig. 7 and that time and temperature increase toward right.

State of Stress:

The state of stress is determined by the magnitude of the stresses in the principal directions. For combined stress or multiaxial stress, the flow stress in one direction will be influenced by the stresses in the principal directions (**28, 30, 31**). The geometry of the part may cause multi-axial stressing, although it may be loaded in only one direction. For example, in a notched specimen loaded only in the axial direction the ratio of the stresses in the principal directions can be varied by changing the geometry of the notch; the flow or fracture stresses will, therefore, also vary (**32**).

CONCLUSIONS

The data presented here and the foregoing discussion can be summarized as follows:

1. The shape of the stress-strain curve at extremely rapid rates of strain at 1200 and 1500 F. of these gas turbine alloys is similar to that at short-time tensile rates. In general, these materials strain harden nearly up to fracture; that is, the load increases with strain and the strain distribution is uniform practically up to fracture.

2. In general, the ultimate strength for wrought alloys at 1200 F. increases with rate up to 10^2 or 10^3 per hour and then decreases. For the cast alloys at 1200 F., the flow stress increases up to a rate of 10 per hour, decreases slightly up to 10^3 per hour and them continues to increase with increasing rate. At 1500 F. the ultimate strength of all the alloys increases with strain rate and shows a slight dip at a rate of about 10^3 per hr.

3. The cast alloys have lower strengths than the wrought alloys at 1200 F. except at the highest strain rates.

4. For a rate of 10 per hour the strengths of all the alloys tested at 1500 F. were within 16 per cent of one another.

5. At a rate of strain of 7.2×10^5 per hour Refractaloy alloy at 1500 F. has the highest strength of all the alloys tested at 1500 or 1200 F.

6. Relative superiority of these alloys depends on the rate of strain at which they are compared.

7. The elongation at fracture at 1200 F. for the gas turbine alloys follows a similar pattern as the "stress curve." At 1500 F., the elongation curve for some alloys does not follow as regular a pattern.

8. The elongations at intermediate and high rates of strain are much greater than that for the creep range by as much as 100-fold.

9. The more desirable working speed at a given temperature can be estimated from the elongation and ultimate-stress curves as a function of rate of strain. This speed is near the peak of the elongation *versus* strain-rate curve.

10. The possible error in the use of rapid methods of sorting or comparing alloys for service in the creep range is pointed out. These sorting methods, however, will give accurate data for processes involving a comparable rate of strain.

11. The relative endurance strengths of a group of similar gas turbine alloys can be estimated from rapid constant-strain-rate tests.

12. A characteristic flow-stress curve is presented which qualitatively expresses the variation in stress as a function of rate of strain, time to failure, or temperature.

13. The characteristic curve, in gen-

eral, is a family of curves for various strains; this family converges to a narrow band for the lower rates or for high temperatures.

14. The factors which influence the characteristic curve are discussed. These factors are (*1*) strain, (*2*) rates of strain and loading or time to failure, (*3*) temperature, (*4*) metallurgical state, and (*5*) state of stress.

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DISCUSSION

MR. J. R. LANE.[1]—I have a question about the shape of the curves. I noticed that there was a knee and I wonder if it could correspond to a change in the type of fracture. It is well known that the strain rate can influence the type of fracture in the same way temperature can, that low strain rates would be conducive to a fracture through the grain boundaries, and conversely, high strain rates would be apt to cause fracture across the grains.

MR. P. R. TOOLIN.[2]—I should like to compliment Mr. Manjoine on his paper and to corroborate his table of fatigue ratios. We have made tension tests and fatigue tests at 1200 and 1500 F. on specimens from the same bar stock for a few high-temperature alloys. Using strain rates, for the tension tests, of the same order of magnitude as that obtained by assuming a saw tooth wave form for the loading cycle in the fatigue test, it appears that the fatigue ratio in general falls between 0.4 and 0.6. It should be noted that while the tension tests were axial, in a machine designed by Mr. Manjoine, the fatigue tests were repeated bending tests (at 7200 cpm.) and that the fatigue ratio is based on the fatigue strength at 100 million cycles.

However, it should be kept in mind that we are dealing with high temperatures and comparing a long-time test with a short-time test. Thus, should an alloy have poor oxidation resistance or be metallurgically unstable at the test temperature, it does not seem impossible that the fatigue ratio might fall outside of the above range. Even at 100 million cycles, some alloys have *S-N* diagrams with appreciable slope, for example a 65 per cent Ni-30 per cent Mo-balance Fe alloy has a fatigue strength, in air at 1500 F., of 35,000 psi. at 100 million cycles, and if the fatigue curve were extrapolated to one billion cycles the fatigue strength would be down to 27,000 psi. A consequence of this is that, even at a great number of cycles, the fatigue ratio is a function of the number of cycles.

But, should the designer have no fatigue data available, the use of a fatigue ratio of 0.5 would give an indication of the 100 million cycle fatigue strength of a high-temperature alloy at 1200 to 1500 F. The use of this value should, of course, be tempered by the considerations mentioned previously.

MR. M. J. MANJOINE (*author's closure*).—Mr. Miller's comments which confirm this type of a generalized flow-stress curve are appreciated.

In reference to Mr. Lane's question, in general, any metallurgical change of the material will result in an anomaly in the flow-stress curve. If the mechanism of flow or fracture changes, then a "knee" will occur in the flow-stress curve. This change is probably a result of the influence of rate of strain on the metallurgical state. There are, however, types of metallurgical changes which do not change the type of failure but which give a knee in the flow-stress curve. Annealing is one of these. Cold-rolled copper at temperatures below the normal recrystallization temperature gives a sharp knee at low strain rates where a long time is necessary to reach a given strain and annealing will occur.

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For these high-temperature alloys, several types of changes take place such as the phase change which occurs in the precision-cast alloys. This change occurs rapidly at the normal service temperatures, and its effect can be seen in the flow-stress curves obtained from tests of most of these alloys.

For the wrought alloys that can be precipitation-hardened a knee is obtained. The change for the alloys reported here has been reduced since they were heat treated to give maximum hardness. However, under the influence of stress and strain additional precipitation occurs.

We are grateful for Mr. Toolin's remarks substantiating our results on fatigue ratio.

THE SELECTION OF A LIMITED NUMBER FROM MANY POSSIBLE CONDITIONING TREATMENTS FOR ALLOYS TO ACHIEVE BEST COVERAGE AND STATISTICAL EVALUATION*

By J. M. Cameron[1] and W. J. Youden[1]

Synopsis

Stress-rupture data present special problems because the number of conditioning factors preclude exhaustive exploration of all combinations of these factors and the test results necessarily involve different test temperatures and loads so that the hours to rupture of specimens from different conditioning treatments are not directly comparable. This paper gives a method for comparing test results and considers the planned selection of combinations of conditioning factors for testing with a view to facilitating the statistical analysis.

During the war, Dr. Gunther Mohling of the Allegheny-Ludlum Steel Corp. obtained stress-rupture data on over 200 specimens of S-816 alloy. About 20 different conditioning treatments were used, the treatments being varied to produce alloys that would satisfy particular demands of production. The Statistical Engineering Laboratory at the National Bureau of Standards was asked to examine the data by statistical procedures to confirm or extend conclusions derived by Dr. Mohling and in the hope of extracting additional information from these measurements on rupture times. Since the data did not represent a planned study of the several conditioning factors involved, the analysis presented a difficult statistical task, but the effort seemed warranted in view of the importance of these alloys.

The first part of the paper discusses the interpretation of the test results and shows why these records present such a difficult problem for statistical analysis. The statistical analysis confirmed Dr. Mohling's conclusions but did not extend them. In the second part of the paper the general problem of exploratory investigations is considered together with ways and means of programming work to facilitate statistical analysis. This leads to the examination of methods of holding the total number of tests down to the minimum number consonant with the objectives of the study.

Conditioning Treatments and Testing Conditions

Stress-rupture measurements were available on 215 specimens of S-816 alloy. These came from 41 heats having similar chemical compositions. Before being tested, the specimens were raised to a solution temperature and held there for a period of time, water-quenched, and then aged for a number of hours at a temperature somewhat

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below the solution temperature. The different solution temperatures, solution times, aging temperatures and aging times used in the conditioning treatments of the specimens are shown in Table I. Not all of the possible combinations of the four factors were used.

The test conditions were not the same for all specimens. Four test temperatures (1200 F., 1350 F., 1500 F., and 1600 F.) and 26 different loadings (ranging from 8000 psi. to 110,000 psi.) were represented. Thus a result from a particular heat and conditioning treatment requires three numbers to characterize it: the test temperature, the load, and the number of hours to rupture.

TABLE I.—LEVELS OF THE FOUR FACTORS USED IN CONDITIONING TREATMENTS.

Solution Temperature, deg. Fahr.	Solution Time, hr.	Aging Temperature, deg. Fahr.	Aging Time, hr.
2150[a]	$\frac{1}{2}$	1350	2
2200	$\frac{3}{4}$	1400[a]	4
2225	1[a]	1500	6
2250[a]	2	1600	8
2275	...	...	16[a]
2300[a]	...	...	19
2325	...	...	...
2350	...	...	...

[a] Levels most often employed.

An attempt was made to compare different solution and aging treatments with the idea of locating the combination of these factors which was associated with best performance as measured by time to rupture. However, when duplicate specimens which had been held at (*a*) different solution temperatures for the same time, (*b*) at the same solution temperature for different intervals, or (*c*) given different aging treatments, were compared, an acute problem arose in that the specimens were often placed under different loads so that the time to rupture cannot be used to compare the performance of the alloy when given different treatments. Not only were different loads used but, as a further complication, the trials were conducted at several test temperatures.

The complexity of the testing procedure, which requires three numbers—the test temperature, the load, and the observed time—to characterize the test results came about from the desire to approximate the conditions under which the alloy was to be used. At the time the tests were conducted, the problem was to produce alloys to meet a particular need, and not for the purpose of a comprehensive study of the factors.

METHOD OF RATING PERFORMANCE

It was decided to start out by making comparisons only in those cases where the tests had been conducted at the same temperature. This still leaves the problem of choosing between two specimens one of which ruptures in say 900 hr. at 20,000 psi. while the other gives way in 500 hr. at 30,000 psi. The non-metallurgist is not in position to know which is better and needs some simple rating scheme.

It was noted that at a given test temperature, for heats with the same conditioning treatment, the product of the load by the logarithm of the time to rupture was approximately a constant. After some trial, all results were assigned a z rating computed from the formula

$z = (10^{-4} \text{ psi.})^k \log(\text{time to rupture})$

where k varies for different temperatures. By trial and error it was found that if the exponent k is taken as 1800 divided by the test temperature the value of z is nearly independent of the load. The z values were found to be in close agreement when specimens from the same heat were tested at the same test temperature but under a wide range of load. The z function was used to convert the times to rupture to a constant load. An example follows:

Heat No. 41592, Test Temperature 1500 F.

Load, psi	Time to Rupture, hr.	z	Calculated hr. to rupture for 22,500 psi.
20 000	494	6.189	218
22 500	262	6.401	262
25 000	121	6.388	232

The value of z depends on the test temperature, but within a test temperature may be used to compare the different methods of conditioning. For each test temperature all results were converted into the number of hours to rupture for the most frequently used loading at that temperature.

TEST RESULTS

When test specimens from different heats were tested, the agreement among the results was nearly as good as when the specimens came from the same heat. It was concluded therefore that the differences in the chemical composition among the 41 heats were of relatively minor importance, and this factor is ignored in the following discussion. Table II shows the average time in hours to rupture for all specimens at each of the conditioning treatments; the number in parenthesis is the number of specimens upon which the average is based. Only aging temperatures of 1400 F. and 1500 F. have been considered, and both these were for 16 hr. (This leaves out 19 specimens aged at 1600 F. which were tested at 1600 F., and 12 specimens given other aging treatments. Neither of these two sets of data would affect the analysis that follows, nor do they display anything by

TABLE II.—AVERAGE NUMBER OF HOURS TO RUPTURE OF SPECIMENS OF S-816 ALLOY GIVEN VARIOUS SOLUTION AND AGING TREATMENTS.

(Number in parenthesis denotes number of specimens on which average is based.)

16 hr. at Aging Temperature, deg. Fahr.	Solution Temperature, deg. Fahr.	Average Time to Rupture, hr.											
		Tested at 1200 F., 60,000 psi.[a]				Tested at 1350 F., 40,000 psi.[a]				Tested at 1500 F., 22,500 psi.[a]			
Solution time		⅓ hr.	⅔ hr.	1 hr.	2 hr.	⅓ hr.	⅔ hr.	1 hr.	2 hr.	⅓ hr.	⅔ hr.	1 hr.	2 hr.
1400	2150	152 (2)	166 (2)	139 (4)			49 (1)	87 (20)				128 (10)	
1500	2150												
1400	2200			126 (2)				42 (2)					
1500	2200											50 (1)	
1400	2225											84 (1)	
1500	2225												
1400	2250			142 (10)				108 (2)				131 (10)	
1500	2250												
1400	2275										167 (1)	118 (1)	
1500	2275												
1400	2300			230 (24)	139 (1)	100 (2)		102 (17)	72 (2)	186 (6)		192 (39)	120 (1)
1500	2300											84 (4)	86 (1)
1400	2325												
1500	2325											76 (1)	
1400	2350							147 (10)					
1500	2350											223 (7)	

[a] When other loads were used times were converted to standard load.

themselves.) The data at the different test temperatures cannot be combined.

INTERPRETATION OF DATA

The effect of solution temperature on the number of hours to rupture is found by comparing specimens which were treated alike in all other respects. The two available sets are shown in Table III, one of which has results for all three test temperatures. These data display a fairly consistent upward trend with increase in the solution temperature at all three test temperatures for the first conditioning treatment of 1 hr. at solution temperature and aging for 16 hr. at 1400 F. This trend is repeated for the second conditioning treatment in which the aging temperature is raised to 1500 F. Thus for both of the two different conditioning treatments higher solution temperatures are showing up best. Generalization of this statement to other conditioning treatments is subject to the usual caution.

TABLE III.—EFFECT OF SOLUTION TEMPERATURE, DURATION ONE HOUR, ON TIME TO RUPTURE.
(Entries show average times in hours and, in parenthesis, the number of specimens.)

Solution Temperature, deg. Fahr.	Specimens Aged 16 hr. at 1400 F.			Specimens Aged 16 hr. at 1500 F.
	Tested at 1200 F., 60,000 psi.[a]	Tested at 1350 F., 40,000 psi.[a]	Tested at 1500 F., 22,500 psi.[a]	Tested at 1500 F., 22,500 psi.[a]
2150	139 (4)	87 (20)	128 (10)	
2200	126 (2)	42 (2)	84 (1)	50 (1)
2225				
2250	142 (10)	108 (2)	131 (10)	
2275			118 (1)	
2300	230 (24)	102 (17)	192 (39)	84 (4)
2325				76 (1)
2350		147 (10)		223 (7)

[a] When other loads were used times were converted to standard load.

TABLE IV.—EFFECT OF SOLUTION TIME ON HOURS TO RUPTURE: ALL SPECIMEN AGED 16 HR. AT 1400 F.
(Entries show average times and, in parenthesis, the number of specimens.)

Solution Time, hr.	Solution Temperature 2150 F.		Solution Temperature 2275 F.	Solution Temperature 2300 F.		
	Tested at 1200 F., 60,000 psi.[a]	Tested at 1350 F., 40,000 psi.[a]	Tested at 1500 F., 22,500 psi.[a]	Tested at 1200 F., 60,000 psi.[a]	Tested at 1350 F., 40,000 psi.[a]	Tested at 1500 F., 22,500 psi.[a]
$\frac{1}{2}$	152 (2)				100 (2)	186 (6)
$\frac{2}{3}$	166 (2)	49 (1)	167 (1)			
1	139 (4)	87 (20)	118 (1)	230 (24)	102 (17)	192 (39)
2				139 (1)	72 (2)	120 (1)

[a] When other loads were used times were converted to standard load.

Table IV shows the effect of varying the solution time at three different solution temperatures. There is an indication of a trend toward better results for the shorter time periods (1 hr. or $\frac{2}{3}$ hr.) at all the indicated solution temperatures. Without knowledge of the missing values in the table, it cannot be stated whether $\frac{2}{3}$ hr. or 1 hr. is the better solution time. Further there is no assurance that a different pattern would not be displayed for different aging treatments.

The data are assembled in Table V to contrast the two aging temperatures of 1400 F. and 1500 F. for 16 hr. It appears that 1400 F. is the better aging temperature.

It is clear that one of the chief difficulties in analyzing these data, apart from the problem of the different testing conditions, arises from the fact that of the 64 possible conditioning treatments (ignoring aging time) comparable data, that is, at a given test temperature, is available on no more than 13 different treatments, and comparisons among these are limited because the times and temperatures used exhibit no systematic pattern.

The remainder of the paper takes up the problem of choosing among the various possible conditioning treatments those which will most efficiently lend themselves to the several comparisons which have been attempted with the data in hand.

TABLE V.—EFFECT OF AGING TEMPERATURE ON NUMBER OF HOURS TO RUPTURE.

(Solution temperature for all specimens was 2300 F. All aged for 16 hr. Specimens were tested at 1500 F. and 22,500 psi. or converted to this load. Entries show average times and, in parenthesis, the number of specimens.)

Aging Temperature, deg. Fahr.	Solution Time, 1 hr.	Solution Time, 2 hr.
1400........	192 (39)	120 (1)
1500........	84 (4)	86 (1)

EXPERIMENTAL PROCEDURES WITH TWO FACTORS

The determination of the optimum levels for just two factors, such as temperature and time, is analogous with mapping an area in search of its highest point. To be certain of finding the optimum combination it is necessary to investigate all possible combinations of several levels for each of the two factors just as it is necessary to take the altitude at every point on a rectangular grid covering the area surveyed in order to find the highest point. A complete investigation in this manner is very often a costly and time-consuming undertaking.

For this reason, in a study of time and temperature, an investigator may select one time period and try runs at different temperatures. Having found the "best" temperature *for this time*, a series of time periods are run holding the temperature constant at this "best" value. In this way an optimum time for this temperature is obtained. In the absence of any other information, these values for time and temperature found in this manner are considered the best combination. By carrying through the analogy with the survey of a land area it is evident that this procedure does not guarantee that the optimum combination (or highest point) will be located.

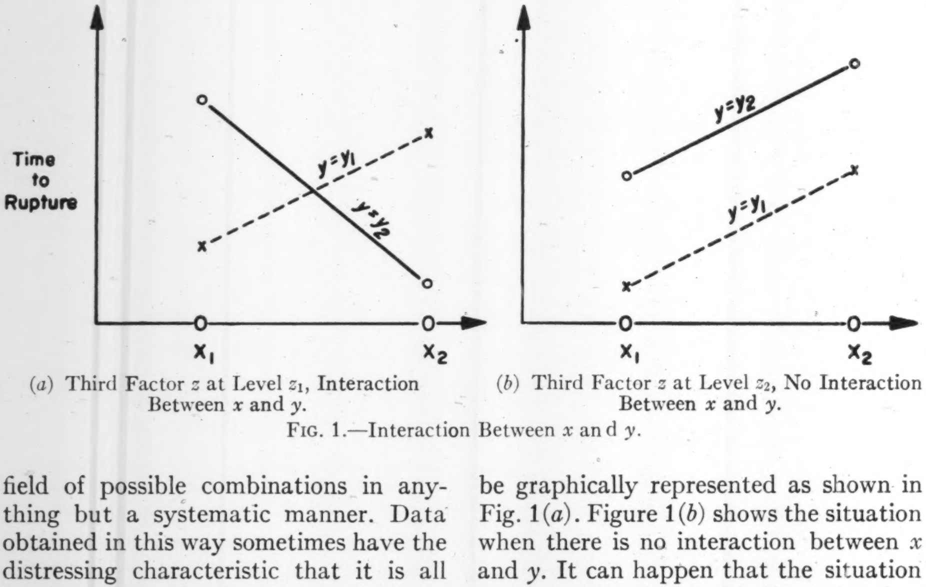
The above simplified procedure assumes that the best temperature for an arbitrarily chosen time period is also the best temperature at all other time periods. There is the possibility that an increase in the temperature is beneficial at one aging time but without effect or even detrimental at some other aging time. In other words the effect of a change in one factor depends on the setting of the other factor. Statisticians have a name for this state of affairs. They speak of such a phenomenon as an interaction because the factors are interacting with each other instead of producing effects which are independent and additive.

Another experimental approach is to select combinations of time and temperature either more or less blindly or in the general vicinity of current practice. This method would require our mythical surveyor to parachute down at a number of points and determine the height at each point. There is only a small chance that the actual optimum combination or the highest point on the field will be found in this way, and even if the highest value found is really the optimum point it is usually impossible to prove that this is so.

This method has one advantage over studying the factors separately, since unsuspected favorable combinations may turn up. For example, if at the usual aging time the higher aging temperatures produce poor results, this may lead to ruling out these higher aging temperatures, whereas it may happen that the higher temperatures are superior at shorter than usual time periods.

EXPERIMENTAL PROCEDURES WITH MANY FACTORS

The problems and difficulties encountered with just two factors are literally multiplied by the inclusion of a third or additional factors. The multiplicity of choices often leads to jumping about the field of possible combinations in anything but a systematic manner. Data obtained in this way sometimes have the distressing characteristic that it is all but impossible to make meaningful comparisons among the results. That is, two results obtained at different temperatures may also differ in other respects, and it is, of course, impossible for anyone, statisticians included, to deduce which factor (or factors) accounts for the observed difference in performance. What is really needed is a guide to the selection of combinations to be tried which will maximize the number of meaningful comparisons for the amount of work done. It would be desirable to arrange matters so that a given result may be compared with several others in turn. This is possible if every combination is tried. As might have been suspected, the most fruitful selection, if a selection has to be made, involves a certain degree of symmetry in the program.



(*a*) Third Factor z at Level z_1, Interaction Between x and y.

(*b*) Third Factor z at Level z_2, No Interaction Between x and y.

FIG. 1.—Interaction Between x and y.

INTERACTIONS BETWEEN FACTORS

It is the possible presence of interactions between the experimental factors that constitutes the core of the problem. An interaction between two factors may be graphically represented as shown in Fig. 1(*a*). Figure 1(*b*) shows the situation when there is no interaction between x and y. It can happen that the situation shown in Fig. 1(*a*) holds if a third factor z is at a particular level z_1, and the interaction either disappears (Fig. 1(*b*)) or is altered at some other level, z_2. If the degree of interaction between two factors x and y is influenced by a third factor, this is described as a second order interaction. Obviously this complicates things, especially if it is now generalized to four or more factors. Fortunately there has now accumulated a considerable body of experimental data where complete and exhaustive explorations of all possible combinations of several factors have been tried. These experiments show that, if as many as four factors are involved, the effect of the four-factor interaction may be ignored, and that, on the average the interaction of three factors is of small importance compared with interactions of two factors. This amounts to saying that as sharp a contrast as shown between the diagrams in Fig. 1 is relatively rare, that usually the relationships shown in Fig. 1(*a*) for example, will be, in the main, reproduced in Fig. 1(*b*). If this assumption can be made, a considerable saving may be effected in the number of combinations which have to be run to disclose not only the primary effect of the factors separately upon the performance, but to establish the existence of interactions (as in Fig. 1(*a*)) between pairs of factors.

			A_1				A_2			
			B_1		B_2		B_1		B_2	
			C_1	C_2	C_1	C_2	C_1	C_2	C_1	C_2
D_1	E_1	F_1	•			•		•	•	
		F_2		•	•		•			•
	E_2	F_1		•	•		•			•
		F_2	•			•		•	•	
D_2	E_1	F_1		•	•		•			•
		F_2	•			•		•	•	
	E_2	F_1	•			•		•	•	
		F_2		•	•		•			•

FIG. 2.—Recommended Initial Sub-set in Exploratory Investigation of Six Factors Each at Two Levels.

What this boils down to is an important point quite overlooked in most experimental studies. It was mentioned earlier that a more or less erratic trying of various combinations leads to results that cannot be matched up for comparisons. By assuming the absence of complicated interactions, it becomes possible to make certain comparisons among the results which would almost surely not be attempted because the balancing out of the factors and two-factor interactions is not at all obvious. The relationships are somewhat involved, and it is the function of the mathematician to indicate the mathematically best selection of combinations and to list the permissible comparisons.

SELECTION OF TREATMENT COMBINATIONS TO BE STUDIED

In an investigation involving several variables, say six factors each at two levels there are in all 2^6, or 64 treatment combinations. These are shown in Fig. 2. If it can be assumed that the three-factor and higher order interactions are negligible, a selection of just half of these treatment combinations can be made that will make it possible, (*a*) to determine the relative importance of the several factors, (*b*) to ascertain the extent to which the effect of a given experimental factor is altered by changing the level of any other factor, and (*c*) to make an estimate of the accuracy of these comparisons. The best selection of 32 out of the 64 is shown by the starred cells in Fig. 2.

			A_1				A_2			
			B_1		B_2		B_1		B_2	
			C_1	C_2	C_1	C_2	C_1	C_2	C_1	C_2
D_1	E_1	F_1	I	III	III	I				
		F_2	III	II	II	III	I			I
	E_2	F_1		I	I					
		F_2	II			II		I	I	
D_2	E_1	F_1					I			I
		F_2	I			I				
	E_2	F_1						I	I	
		F_2		I	I					

FIG. 3.—Example of Three Stage Investigation of Six Factors Each at Two Levels.

Inspection of the starred entries in the diagram reveals that 16 runs have been made at the level A_1 of factor A, and that these are matched by 16 other runs with A at level A_2. The matching is a bit involved as will be apparent from scrutiny of the four selections in the top line, two of which are at level A_1 and the other two at level A_2. These four are identical as far as factors D, E, and F are concerned. But the factors B and C are not used in the same combinations for A_1 as for A_2. This state of affairs exists in every row and makes it possible to take the average of the 16 runs on the left half and contrast this with the average of the 16 on the right in order to detect, with all the power that an average of 16 gives, whether or not there is some small persistent advantage in one level of A over the other level.

The above comparison of A_1 and A_2 ignores the question of the interacting effect of other variables. This effect may be revealed by noting that of the 16 runs at each level of A there are eight with the variable D at D_1 and eight at D_2. (This is also true for every pair of variables.) The averages may therefore be taken over these sets of eight runs to form the following four averages:

	A at A_1	A at A_2
D at D_1	A_1D_1	A_2D_1
D at D_2	A_1D_2	A_2D_2

The comparison of A_1 and A_2 at D_1 gives

$$A_1D_1 - A_2D_1 = A_{D1}$$

and at D_2,

$$A_1D_2 - A_2D_2 = A_{D2}$$

If the difference between A_1 and A_2 depends on D, this is revealed by the difference between A_{D1} and A_{D2}. Taking this difference is equivalent to combining the sets A_1D_2 and A_2D_2 and comparing with the combined sets A_2D_1 and A_1D_2. This comparison, called the interaction of A and D, uses all the data and is analogous to the direct comparison of the two levels of A.

TABLE VI.—SELECTION OF 81 TREATMENT COMBINATIONS FROM THE 243 COMBINATIONS OF FIVE FACTORS EACH AT THREE LEVELS.

Aging temperature			1400 F.			1500 F.			1600 F.		
Aging time, hr.			12	16	20	12	16	20	10	16	20
Factor F	Solution Temperature, deg. Fahr.	Solution Time, hr.									
F_1	2250	½	*	*	*						
		1				*	*	*			
		2							*	*	*
	2300	½							*	*	*
		1	*	*	*						
		2				*	*	*			
	2350	½				*	*	*			
		1							*	*	*
		2	*	*	*						
F_2	2250	½				*	*	*			
		1							*	*	*
		2	*	*	*						
	2300	½	*	*	*						
		1				*	*	*			
		2							*	*	*
	2350	½							*	*	*
		1	*	*	*						
		2				*	*	*			
F_3	2250	½							*	*	*
		1	*	*	*						
		2				*	*	*			
	2300	½				*	*	*			
		1							*	*	*
		2	*	*	*						
	2350	½	*	*	*						
		1				*	*	*			
		2							*	*	*

By a similar argument, regroupings of the data lead to estimates of interactions involving three variables. In this experimental arrangement it is assumed that these interactions are negligible and they are used as a yardstick to judge the significance of the comparisons discussed above. These computations are explained under the title "Analysis of Variance" in statistical texts.

A similar relationship holds for any other factor due to the inherent symmetry of the selection. If only one fourth of the 64 combinations are to be used the best selection is shown by the cells with Roman I in Fig. 3.

This surveys the territory as well as can be done with this limited number of runs. It is obvious that with only this small fraction of the total number of treatment combinations, conclusions will be more limited and less authoritative. Nevertheless such an exploratory survey may serve to narrow down the field of investigation to manageable dimensions so that sufficiently intensive examination may be conducted therein. Thus this survey may show that the levels A_1 and D_1 are showing up best, and the question is still open on the other variables. The cells with Roman II indicate a logical second phase which may suffice to settle the issues or at least to settle the matter on one of the variables, say E, in which case four more runs as shown by the cells with Roman III when combined with the prior runs constitute an intensive survey of these remaining three factors.

It is obvious that there has been a marked saving by this step-wise procedure, but this saving is only going to be possible provided the program is thought out in advance. Finally, it is clear that having used this procedure to single out the important factors, these particular factors can then be tried at additional levels at relatively small expenditure of effort.

Five factors, each at three levels, yield 243 different combinations. From these, 81 treatment combinations may be selected which permit appraisal of the performance of the levels of the different factors. The selection of treatment combinations to be used are starred in Table VI. This design comes from a paper by D. J. Finney.[2]

Many other investigations besides the alloy study discussed in this paper also involve the same problem of an immense number of runs. Very often the available laboratory or engineering facilities place a ceiling on the number of runs which can be made. This forces some selection, and the determination of the particular runs to be tried depends very largely upon the exercise of good engineering judgment. The best results will be obtained when this judgement is combined with the knowledge that certain selections do make particularly efficient use of the data. The mathematical solution of this problem is a very recent development. Reports of the application of this technique are just beginning to appear in the literature and these indicate that it is going to be a valuable tool in the programming of extensive investigations of the characteristics and performance of new materials.

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[2] D. J. Finney, "Recent Development in the Design of Field Experiments: III Fractional Replication," *Journal of Agricultural Science*, Vol. 36, Part 3, pp. 184–191 (1946).

DISCUSSION

Mr. Leo Schapiro.[1]—The relationship described by the authors—between the stress to rupture, hours to rupture, and temperature—is indeed very interesting. The reduction of all test data for this S-816 material to a semilogarithmic formula enables the prediction of results within the limits of the variables making up the formula. The results of such a prediction are here described.

Using the authors' values for z for the temperatures 1200, 1350, 1500, and 1600 F., Fig. 4 is obtained relating z to test temperature. Selecting three temperatures and an applicable stress for each, a time to rupture was calculated, from the authors' formula. Mohling of Allegheny-Ludlum Steel Corp., who furnished the original data, was kind enough to make the three tests to check the predictions. The test conditions, the predicted life time, and the actual life time are given below:

Test Temperature deg. Fahr.	Stress, psi.	Predicted Rupture Time, hr.	Actual Rupture Time, hr.
1250	50 000	152.5	595+
1450	30 000	77.5	69.5
1550	20 000	62.5	58

Mr. J. G. Thompson.[2]—I still am a little suspicious of predictability of high-temperature test results. I cannot forget the great amount of work that was done some years ago in investigating the possibility of accelerated creep tests, in the hope that the results obtained from them could be extrapolated to the conventional long-time creep test results.

Those attempts were not successful, and with that in mind, I feel that the conversion factor which has been demonstrated to exist for this particular set of stress-rupture data, applies to these data only, and within a limited range.

I hope that additional work will demonstrate that a similar conversion factor can be found for other sets of data. Perhaps eventually we can disprove the old creep theory, and there may be some relation developed which will hold for all cases.

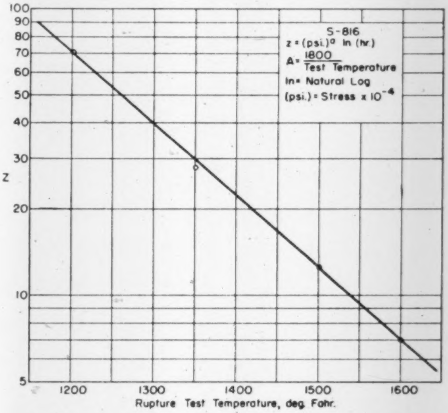

Fig. 4.—Relation of z to Test Temperature.

Mr. H. C. Amtsberg.[3]—The authors have given, I believe, the mathematical approach, that is, the approach of the mathematician to the unscrambling of high-temperature alloy data, and I certainly commend them for that effort.

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[1] Chief Metallurgist, Materials Lab., Douglas Aircraft Co., Inc., Santa Monica, Calif.

[2] Chief, Division of Metallurgy, National Bureau of Standards, Washington, D. C.

[3] Manager, Metallurgical Engineering, Materials Engineering Dept., Westinghouse Electric Corp., East Pittsburgh, Pa.

However, I doubt that that is the easy way to do it. I think the mathematician somewhat resents the approach of the physical or mechanical metallurgist who obtains an enormous quantity of data, hands it to the mathematician and says "Please unscramble this for me." It is a tough proposition.

I believe the authors, to some extent, are making a plea to the metallurgist, in particular, that he review the program with the mathematician before running his tests, so that significant data are obtained, and that the maximum amount of information can be obtained from the laboratory data.

At Westinghouse, we have taken somewhat the opposite approach. We thought it might be easier to make reasonably good statisticians out of metallurgists by giving them the formulae and having them analyse the data. That has worked out quite well, we think, but it has certainly revealed the extremely broad variability of high-temperature properties. I shall attempt to illustrate this, at the possible expense of laboring some simple statistics—and I want you to remember that I am not a statistician—I am alleged to be a metallurgist, of some sort.

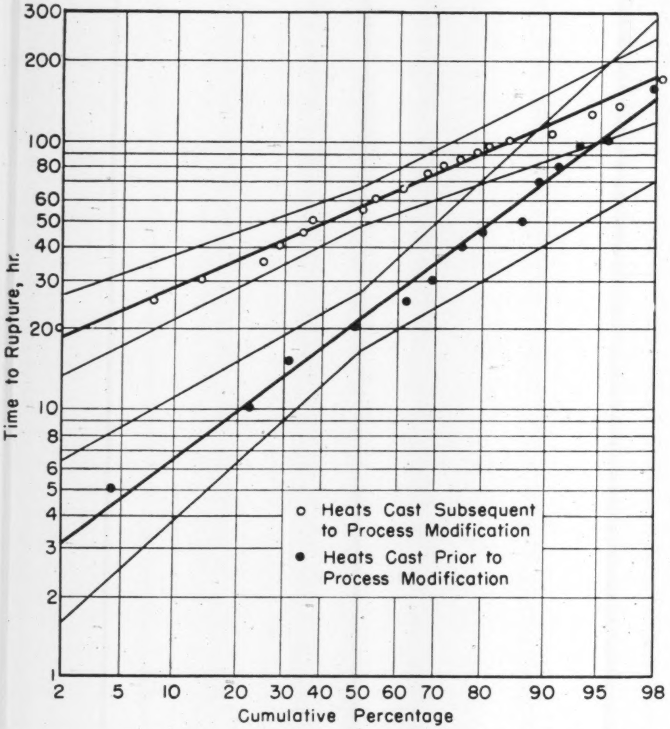

FIG. 5.—Comparison of Creep Rupture Life of Stellite 31 (AMS 5382) Tested at 1500 F. and 30,000 psi.

Heats before and after changes in melting practice.

If the percentage axis of a cumulative frequency distribution curve is deliberately skewed symmetrically about the 50 per cent line with a log scale, we have what is known as probability paper on which a normal distribution plots as a straight line. On this probability paper, the average value is that which falls at the 50 per cent ordinate. The standard deviation (sigma = σ) is the difference between the 50 per cent ordinate and either the 16 or 84 per cent ordinate intersection with the curve. This system is at once a simple but extremely powerful statistical tool.

Of course, anyone familiar with statistics understands that a finite number of observations does not necessarily infer the true distribution with absolute certainty. However, the area or envelope in which the true distribution lies with a given probability can be calculated with ease. Such distribution curves with their probability envelopes (95 per cent certainty level) are shown in Fig. 5.

The broad range over which rupture time varies for an alloy, even with all manufacturing variables held as constant as possible, becomes immediately obvious. The variation between the 2 and the 98 per cent properties is at least an order of magnitude. We find that this same order of variation appears to hold for all high-temperature properties of alloys, cast or wrought. Even when a number of samples have been taken from one piece of bar stock of an alloy and given one treatment, the variability is at least a part of a magnitude, say from 10 to 35 hr. for rupture time.

Figure 5 illustrates very nicely how this method of analysis can be used to determine if a truly significant improvement has been brought about by a process change, as has been the case here. The mean properties have been raised, but more importantly, the process modification has increased the uniformity as indicated by the decreased slope of the curves. The greatest change therefore has been in the minimum value—all-important from the design and service standpoint.

I should like to ask the authors if the data employed in their mathematical study have been tested in some manner for significance, because we find that an absolute minimum of six and very frequently as many as 15 points are needed to establish a distribution with any reasonable accuracy. Statistical theory tells us that for a given probability, the width of the bands is a function of the variability of the data and the number of tests. For a very few points the probability bands tend to be extremely wide, with considerable overlapping very likely. Thus it may be difficult or impossible to determine to which family a given value belongs. In the accompanying chart there is overlapping above about the 80 per cent line even though these curves are derived from 20 values each. If the first values obtained lay in this region (a distinct possibility) and test work then stopped, grossly misleading conclusions could be drawn. Our experience shows that sufficient data must be obtained and suitably analyzed for a proper selection of alloys to be made.

MR. J. M. CAMERON (*author*).—The relation between time to rupture and the load and test temperature used here was evolved after some trial and error and seemed to fit the data adequately. It is desirable to have such a relationship in order to facilitate the analysis of data obtained under different testing conditions and to effect a reduction in testing since data from one set of conditions can be used to predict performance under diverse conditions. To be valid, the form of the relationship should be derived from theoretical considerations. Since our z value does not have such theoretical support it is not surprising to find on some occasions experimental results that disagree with the predicted value as pointed out by Mr. Shapiro.

I agree with Mr. Amtsberg's remark that the job of reducing a large mass of data can at times be a discouraging one for the mathematician. If some attention is given to the problems that arise in the analysis of large masses of data, prior to the completion of the tests, there will be an opportunity to include those tests which will facilitate the mathematical analysis.

It seems to me that in due time a lot of things which now look to be purely mathematical or statistical will become part of laboratory technique. Experimental designs, patterns, and schedules are properly the concern of the laboratory man.

MAGNESIUM-CERIUM WROUGHT ALLOYS FOR ELEVATED-TEMPERATURE SERVICE*[1]

By K. Grube,[2] J. A. Davis,[2] and L. W. Eastwood[3]

This paper, the second in a series on the development of aluminum- and magnesium-base alloys for elevated temperature, deals with magnesium-cerium wrought alloys for this type of service.

The general aim of the investigation described in this paper was the development of magnesium-cerium alloys having better load-carrying capacity at 600 F. than existing wrought alloys of the EM62, EM42, and EM22 types. All three of these alloys contain approximately 1.6 per cent manganese and 6, 4, and 2 per cent rare earths, respectively.

The development of magnesium-rare earth casting alloys described in a recent paper[4] was coordinated with the parallel study of the wrought alloys. As in the development of magnesium-cerium cast alloys, rare-earth additions rather than cerium were made to magnesium. However, for the sake of convenience, the term "cerium" will be used, though the addition actually was "rare earths" composed of approximately 50 per cent cerium and the balance mainly other metals in the rare-earth group.

Experimental Procedures

The general procedure consisted of melting the alloys, casting them into an extrusion billet, scalping the billet to approximately 3 in. in diameter, extruding the billet to $\frac{3}{4}$ in. in diameter, heat treating the bars, and then performing the following tests:

1. Tension tests at room temperature.
2. Tension tests at 600 F.
3. Short-time creep tests at 600 F.

As will be shown later, more detailed tests were made on an improved alloy selected for further study.

Melting and Casting:

The methods of melting, making the various alloy additions, and casting were the same as those described elsewhere.[4] The metal was poured into $3\frac{1}{2}$-in. diameter steel molds and, in some instances, into the green sand 4-bar test-bar mold, into chill-cast slabs 5 by 5 by $\frac{1}{2}$ in. thick or 8 by 6 by 1 in. thick, and into forging ingot 5 by 5 by 8 in. high. The forging ingot mold consisted of cast iron side walls with a copper bottom and a refractory hot top to insure adequate feeding. This mold, not preheated, was tilted and filled to the top of the refractory riser.

A special water-cooling technique was employed to chill the $3\frac{1}{2}$-in. diameter extrusion billet. The steel ingot mold was preheated to a temperature equal to the pouring temperature of the metal, namely, about 1350 F. The preheated

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] The work described in this paper was sponsored by the Nat. Adv. Commitee for Aeronautics, under Contract NAw-5385 during the period from August 20, 1946, to August 20, 1947, and Contract NAw-5627 during the period July 19, 1948, to July 18, 1949.

[2] Research Engineer, Battelle Memorial Institute, Columbus, Ohio.

[3] Supervisor, Battelle Memorial Institute, Columbus, Ohio.

[4] K. Grube and L. W. Eastwood, "Magnesium-Cerium Cast Alloys for Elevated-Temperature Service," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 989 (1950).

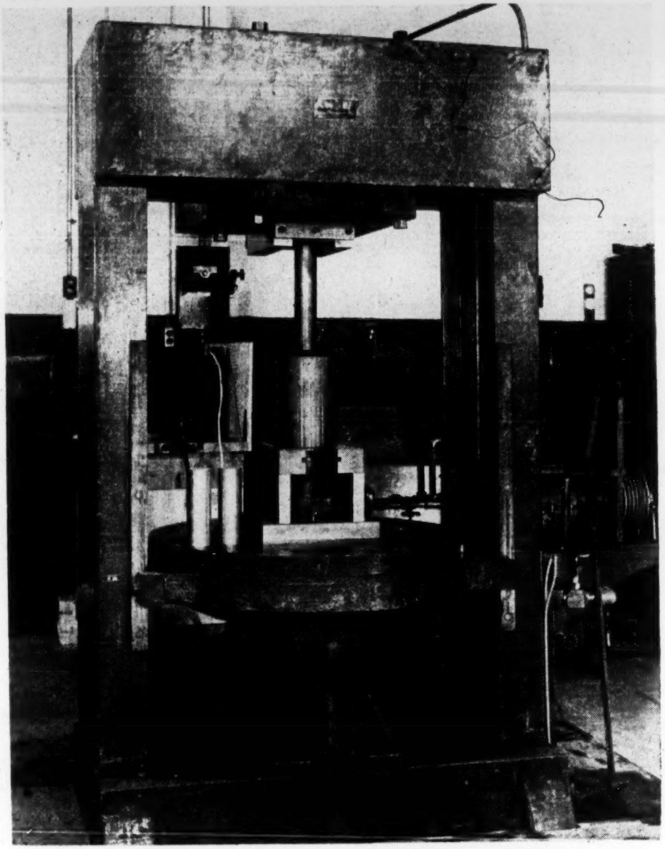
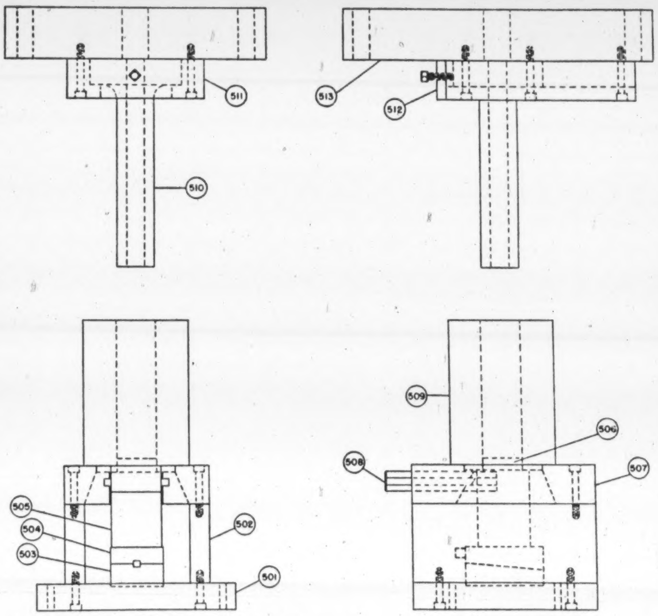


FIG. 1.—General View of Indirect Extrusion Press.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501—Base plate
502—Spacer
503—Tower wedge
504—Top wedge
505—Plug support
506—Cylinder plug
507—Cylinder support plate
508—Stripping block
509—Cylinder
510—Plunger
511—Plunger guide
512—Stop strap
513—Top plate

FIG. 2.—Assembly Drawing of Extrusion Tools.

ingot mold was than tilted and the melt poured down the side with a minimum of turbulence. Because the mold was preheated, it was possible to let certain heavy nonmetallic particles, which may have entered the ingot mold, settle to the bottom of the ingot. Water was then introduced in a tank in which the ingot mold was placed. The water level was raised gradually, during which time the ingot mold was vibrated. In this way, directional solidification from the bottom upwards was obtained. A short piece was cut from the bottom end as well as the top end of the extrusion billet. It was also scalped over the entire surface to $3\frac{1}{16}$ in. in diameter prior to extrusion.

Usually, only the $3\frac{1}{2}$-in. diameter extrusion billet was cast. When test bars were poured, they were heat treated and subjected to tension tests at room temperature and 600 F. and, in some instances, also to creep tests at 600 F. Flat slabs were occasionally poured and then rolled to sheet in order to compare the room-temperature and high-temperature properties of the alloy in the form of sheet. This was done only with compositions of maximum interest.

Heat Treatment and Testing:

Most of the alloys were solution heat treated at 1040 F., cooled in air, and aged at 400 F. prior to test, as described in another paper.[4] The bars tested at 600 F. or at other elevated temperatures were stabilized according to the following schedule:

1. Those tested at 300 F. were placed in the heat-treated and aged state.
2. Those tested at 400 F. were first heat treated and aged, then annealed for 24 hr. at 500 F. prior to testing.
3. Bars tested at 500 F. were first heat treated and aged, and then annealed at 575 F. for 24 hr., prior to testing.
4. Bars tested at 600 F. were heat treated and aged, then stabilized for 24 hr. at 650 F. prior to testing.

Tension tests were made at room temperature and at 600 F. More complete tension tests were conducted on the alloy of optimum composition at 300, 400, 500, and 600 F. The creep test for screening the alloys was conducted at 600 F. under a 2000-psi. load. Creep tests were also run at other loads on the alloy of optimum composition at 600 F. The methods of making the high-temperature tension tests and creep tests are the same as those described elsewhere.[4]

FABRICATION METHODS

A 250-ton press, Fig. 1, was used to make the extrusion by the indirect method. The tools used for this indirect extrusion are illustrated by Fig. 2. Billets $3\frac{1}{16}$ in. in diameter were extruded to $\frac{3}{4}$-in. rod for the experimental tests. The indirect extrusion method was chosen over the direct method for the following reasons:

1. Lower maximum pressures are required, thereby permitting the use of a larger extrusion billet for a given press capacity.
2. The extruded rod is reported to be more uniform from end to end because of the manner in which the metal flows from the container through the die.

When using this method, the extrusion billet was placed in the preheated container, the die at the end of the ram was moved downward, and the metal extruded through the hollow ram up through the open top of the press.

In order to establish the effects of extrusion procedure, a number of extrusion variables were studied, as described later in this paper.

A single-stand, 12-in. experimental rolling mill was employed for rolling some of the experimental alloys. Frequent reheating and light reductions per pass were found to be prerequisites necessary for the production of suitable sheet material.

The forging tests on the 5 by 5 by 8-in. ingot were carried out on an air-driven drop hammer. The dies were not preheated prior to the forging tests. In all probability, press forging would be more practical than the drop forging method employed. However, press forging equipment was not available.

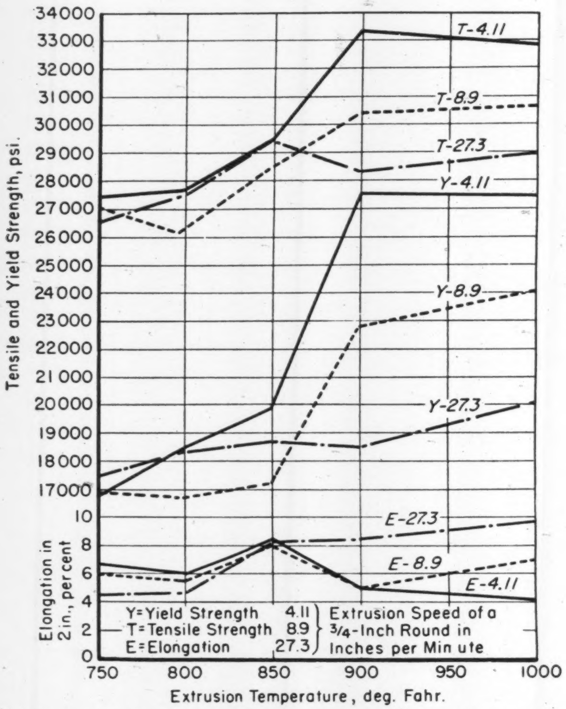
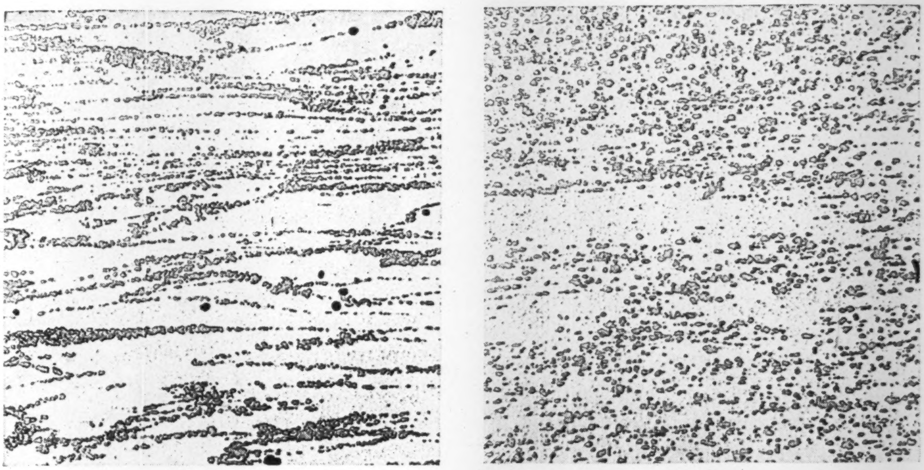


FIG. 3.—Effect of Extrusion Speed and Temperature on Room-Temperature Properties.

EXTRUSION VARIABLES

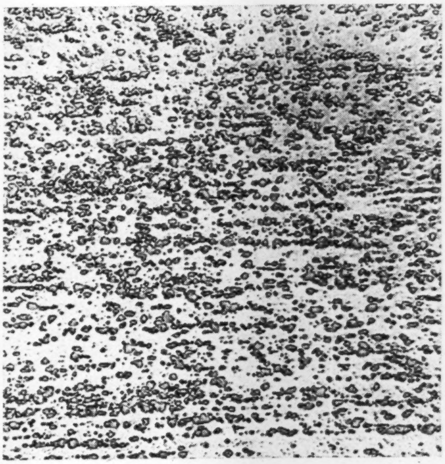
The principal factors affecting the quality of the product of the extrusion process are degree of reduction, rate of extrusion, and temperature of extrusion. The effects of extrusion temperature and extrusion rates of 4.1, 8.9, and 27.3 ft. per sec. are shown by Fig. 3. These data are for EM62 alloy, heat treated 16 hr. at 1040 F. and aged 16 hr. at 400 F. It is quite evident that the lower speeds and higher extrusion temperatures produce the highest tensile strength and yield strength, with somewhat lower elongation.

The effect of the degree of reduction by the indirect extrusion method was also investigated. For this work, EM62 composition was extruded at 800 and 1000 F. through a $\frac{1}{2}$-in. diameter die (37:1 reduction) and a $\frac{3}{4}$-in. diameter die (16:1 reduction), using an extrusion speed of 4.11 in. per min. Some of the extruded material was heat treated 16 hr. at 1040 F. and aged 16 hr. at



(*a*) Center of a Longitudinal Section from a 1- by 1-in. Extrusion. Reduction 7:1, 85.9 per cent.

(*b*) Center of a Longitudinal Section from a ¾-in.-Diameter Extrusion. Reduction 16:1, 93.75 per cent.



(*c*) Center of a Longitudinal Section from a ½-in.-Diameter Extrusion. Reduction 37:1, 97.22 per cent.

FIG. 4.—Microstructure of EM62 Magnesium Alloy After Extrusion Reductions of 85.9, 93.75, and 97.22 per cent. Unetched, as Extruded (×100).

400 F. The rest of the extruded bar was not solution heat treated, but was given only a stabilizing treatment of 24 hr. at 650 F. Tensile properties were obtained at room temperature and at 600 F., and creep tests were also run at 600 F.

As a result of the work, it was found that the tensile properties at room temperature and at 600 F. and the resistance to creep of this alloy at 600 F. were not appreciably affected by the degree of reduction by extrusion in the range of 16:1 and 37:1. Photomicrographs of EM62 alloy given 7:1, 16:1, and 37:1 reductions by the indirect extrusion process are shown by Fig. 4. It will be noted that some improvement in uniformity of the structure occurs when the degree of reduction is increased from 7:1 to 16:1, but relatively little improvement results from the further increase of 16:1 to 37:1.

Standard Extrusion Procedure:

As a result of the study of the extrusion variables, the standard method of extrusion selected for the fabrication of experimental alloys was as follows:

1. A rate of 4.1 ft. per min.
2. A reduction of 16:1, resulting in a $\frac{3}{4}$-in. diameter extrusion.
3. Extrusion temperature about 1000 F. with the container temperatures at 950 to 1000 F.

EFFECTS OF HEAT TREATMENT

The EM62 alloy extruded by the standard conditions outlined above was subjected to tension tests at room temperature and 600 F. and to creep tests at 600 F. The alloy was tested after heat treating for 16 hr. at 1040 F., and aging 16 hr. at 400 F. These specimens tested at 600 F. were also stabilized 24 hr. at 650 F. The effects of the solution-heat-treating EM62 extrusions are shown by the following data:

Property	Stabilized Only	Heat-Treated, Aged, and Stabilized
Tensile strength at room temperature, psi.	31 500	31 500
Yield strength, 0.2 per cent offset, at room temperature, psi.	22 500	25 000
Elongation in 2 in. at room temperature, per cent	5.0	3.5
Tensile strength at 600 F. psi.	9750	11 400
Yield strength, 0.2 per cent offset at 600 F., psi.	5500	7000
Elongation in 2 in. at 600 F., per cent	135	125
Minimum creep rate, 1300 psi. load at 600 F., per cent per hr.	0.01 to 0.03	0.0004

It is quite evident that the solution heat treatment has a very beneficial effect on both the tensile properties and the creep resistance at 600 F. but has little or no effect on the room-temperature tensile properties, at least when the stabilizing treatment of 24 hr. at 650 F. is also applied.

The effects of heat treatment were rechecked on EM42 alloy extrusion as shown by the data in Table I. The data on the room-temperature tensile properties appear to vary somewhat with the different heats. However, solution heat treating has a slightly beneficial effect on the tensile strength at 600 F., relatively little effect on the yield strength at 600 F., but improves the resistance to creep appreciably.

As will be shown later, the creep results on EM62 and EM42 extruded alloys are somewhat erratic. However, because of the above results, solution heat treatment appeared to be beneficial, and all the alloys were heat treated for 16 hr., usually at 1040 F., followed by an aging treatment of 16 hr. at 400 F. The alloys tested at elevated temperature were stabilized prior to test as indicated previously.[4]

ALLOY DEVELOPMENT

After investigating the effects of extrusion techniques, the alloy development was undertaken. The general procedure consisted of first determining the effects of manganese and cerium in order to establish the proper base composition. A systematic study was then undertaken of the effects of various alloy additions to this base. Again, the general procedure was to determine the effects of single additions to this base by obtaining tensile properties at room temperature and at 600 F. and subjecting the experimental composition to the standard creep test consisting of either 1300 psi. or 2000 psi. load applied at 600 F. This test was run approximately one week and then discontinued if the creep resistance was

TABLE I.—EFFECT OF VARIOUS HEAT TREATMENTS ON THE PROPERTIES OF EXTRUDED EM42 ALLOY.

Heat	Analyses, per cent		Heat Treatment[b]	Test Temperature, deg. Fahr.	Tensile Properties[a]				Creep Properties at 600 F.-2000 psi. Load					
	Cerium	Manganese			Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Duration, hr.	Initial Deformation, per cent	Minimum Creep Rate, per cent per hr.	Deformation at End of 120 hr., per cent	Total Deformation, per cent	Contraction on Release of Load, per cent
A4932	4.25	1.51	Aged	70	33 000	38 000	4.0	3.3						
A4932			Aged	600	7 400	10 400	108.5	91.0						
A4931	4.45	1.50	Aged	600					306.0	0.039	0.0045	0.630	1.37	0.010
A4932	4.25	1.51	A&S	70	29 850	35 550	5.5	5.8						
A4930	4.44	1.50	A&S	600	7 800	10 450	133.7	92.7						
A4931	4.45	1.50	A&S	600					98.0	0.042	0.019	.....	1.77	0.050
A4929	4.40	1.49	HTA	70	34 300	37 150	4.1	6.2						
A4928	4.43	1.48	HTAS	600	7 550	11 275	106.3	86.5	170.0	0.042	0.0038	0.495	0.682	0.046
A4931	4.45	1.50	HTAS	600					306.0	0.042	0.00025	0.197	0.312	0.036
A4931	4.45	1.50	HTAS	600					306.0	0.044	0.0014	0.275	0.670	0.042
A5111	4.03	1.61	HTA	70	32 400	37 900	3.5	3.0						Water quench
			HTQAS	600	7 825	11 700	83.7	73.3						
A4929	4.40	1.49	HTQA	70	38 125	41 000	3.5	4.1						0.030 Water quench
A4928	4.43	1.48	HTQAS	600	7 875	11 400	115.0	91.5	305.0	0.034	0.0007	0.268	0.406	
A4928	4.43	1.48	HTQA	70	33 325	38 500	5.0	4.8						0.043 Oil quench
			HTQAS	600	7 800	11 100	146.2	94.7	331.0	0.047	0.00045	0.215	0.317	

[a] Average of two tests.

[b] Aged—Heated for 16 hr. at 400 F. and air cooled.
Aged and Stabilized—Stabilized at 650 F. for 24 hr. after the above aging treatment.
HTA—Solution heat treated at 1040 F. for 24 hr., air cooled, and aged 16 hr. at 400 F.
HTQA—Solution heat treated at 1040 F. for 24 hr., quenched in either water or oil, and aged at 400 F. for 16 hr.
HTQAS—Same as above, but stabilized 24 hr. at 650 F. following the aging treatment.
HTAS—Same as HTA, but stabilized 24 hr. at 650 F. and air cooled, following the aging treatment.

TABLE II.—EFFECT OF MANGANESE ON THE TENSILE PROPERTIES OF EXTRUDED MAGNESIUM-CERIUM ALLOYS AT ROOM TEMPERATURE AND 600 F.

Heat	Manganese, per cent	Room Temperature[a]			600 F.[b]				
		Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation, in 2 in., per cent	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Minimum Creep Rate, per cent per hr.	Total Deformation at End of 120 hr., per cent
				2 per cent Cerium					
A5107	0.0	17 575	28 500	....	4 300	7 275	87	Very high	Failed
A4970	0.81	26 650	32 750	7.4	6 100	9 475	115	0.002	0.27
A5270	0.89	20 625	29 625	14.8	5 900	8 550	128		....
A4971	1.24	28 400	33 250	5.5	7 200	9 850	95	0.002	0.39
A4972	1.55	25 350	31 450	5.5	7 150	9 325	81	0.001	0.30
A4975	1.90	27 250	33 000	7.2	7 400	9 950	116	0.001	0.29
				4 per cent Cerium					
A4847	0.0	29 100	32 350	5.7	4 925	10 125	129	Very high	Failed
A4846	0.62	26 175	30 850	6.5	6 150	10 400	126	0.030	4.7
A4845	1.03	25 250	30 700	5.5	6 575	10 675	125	0.023	4.4
A4844	1.38	24 850	30 250	3.5	6 700	10 800	129	0.016	5.0
A4843	1.66	27 450	32 000	5.2	7 025	11 300	100	0.01	1.3

[a] HTA—Solution heat treated at 1040 F. for 24 hr., air cooled, and aged 16 hr. at 400 F.

[b] HTAS—Same as HTA, but stabilized 24 hr. at 650 F. and air cooled, following the aging treatment.

Load applied to produce a crosshead movement of 0.01 in. per min. per in. of gage length until the 0.2 per cent offset yield was obtained; rate then increased to 0.03 in. per min. per in. of gage length.

not appreciably better than that of the base composition. If it did have appreciably higher resistance to creep, then the test was extended to approximately 300 hr. An attempt was then made to combine those additions which had a beneficial effect in order to obtain still further improvement in the high-temperature properties of the wrought magnesium-cerium alloys.

TABLE III.—EFFECT OF VARIOUS HEAT TREATMENTS ON THE PROPERTIES OF MANGANESE-MAGNESIUM EXTRUDED ALLOYS CONTAINING VARIOUS AMOUNTS OF CERIUM.

Heat	Analyses, per cent		Heat Treatment[b]	Test Temperature, deg. Fahr.	Tensile Properties[a]				Creep Properties–2000 psi. Load					
	Cerium	Manganese			Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Duration, hr.	Initial Deformation, per cent	Minimum Creep Rate, per cent per hr.	Deformation End of 120 hr., per cent	Total Deformation, per cent	Contraction on Release of Load, per cent
A4993.....	0.0	1.80	HTA	70	27 000	30 450								
			HTAS	600	3 400	4 400	86.0	63.0	244	0.041	0.0046	0.680	7.65	0.010
			HTQAS	600					607	0.042	0.0003	0.090	0.281	0.044
A5257.....	0.0	1.84	HTA	70	30 200	35 000	2.5	2.4						
			HTQA	70	23 500	33 750	3.0	3.6						
			HTAS	600	3 175	4 000	92.0	59.0	97	0.052	0.012	.....	4.10	0.030
			HTQAS	600	4 675	5 400	47.0	42.7	553	0.047	0.00046	0.114	2.6[c]	[d]
A5258.....	0.20	1.94	HTA	70	24 900	27 250	1.5	1.7						
			HTAS	600	5 150	8 300	86.5	67.9	566	0.043	0.0004	0.110	2.6[c]	[d]
A5109.....	0.41	1.65	HTA	70	23 900	31 000	3.0[e]	3.1[e]						
			HTAS	600	6 075	9 200	46.0	58.8	599	0.046	0.0020	0.301	1.48	0.040
			HTQAS	600					594	0.041	0.0040	2.92	5.42	0.040
			ANN	600					56	0.049	0.077		5.71	0.050
A5259.....	0.73	1.86	HTA	70	26 625	32 100	3.5	3.9						
			HTAS	600	6 275	8 750	79.2	60.1	336	0.044	0.0022	0.230	0.502	0.044
A5110.....	1.34	1.56	HTA	70	29 900	35 375	5.5	7.5						
			HTAS	600	7 275	10 650	86.0	79.7	597	0.041	0.0014	0.303	1.05	0.050
			HTQAS	600					599	0.043	0.001	0.375	0.952	0.034
			ANN	600					152	0.044	0.057	7.500	9.92	0.070
A5260.....	1.39	1.94	HTA	70	31 500	35 175	7.0	7.3						
			HTAS	600	7 100	9 750	100.0	74.2	609	0.046	0.0019	0.285	1.89	0.030
A4975.....	2.41	1.90	HTA	70	27 250	33 000	7.2	10.6						
			HTAS	600	7 400	9 950	116.0	83.2	621	0.049	0.0008	0.294	0.714	0.027
			HTQAS	600					601	0.050	0.0035	0.500	3.04	.....
			ANN	600					51	0.055	0.175	.....	11.04	0.040
A4974.....	4.31	1.77	HTA	70	31 900	36 150	4.0	5.8						
			HTAS	600	7 725	10 500	91.7	84.5	551	0.043	0.0034	0.520	1.96	0.050
			HTQAS	600					602	0.041	0.0006	0.300	0.675	0.032
			ANN	600					102	0.046	0.060	.....	6.95	0.050
A4973.....	6.01	1.81	HTA	70	27 500	37 500	2.8	3.3						
			HTAS	600	7 750	10 850	122.5	89.7	360	0.042	0.0020	0.504	1.53	0.045
			HTQAS	600					594	0.046	0.00043	0.230	0.445	0.045
			ANN	600					126	0.040	0.077	9.000	9.96	0.040
A5277.....	5.33	1.75	HTA	70	35 250	35 800	3.2	3.5						
			HTAS	600	8 300	11 700	125.5	92.3	458	0.050	0.010	1.130	4.80	0.040
			HTQAS	600					534	0.040	0.00075	0.550	0.899	0.035

[a] Average of two tests.

HTA—Solution heat treated 24 hr. at 1040 F., air cooled, and aged at 400 F. for 16 hr.
HTAS—Stabilized 24 hr. at 650 F. after the HTA treatment.
HTQAS—Water quenched from the solution heat-treating temperature. Aged and stabilized as above.
ANN—Furnace cooled from solution heat-treating temperature after soaking for 24 hr.

[c] Measured after rupture.

[d] Specimen broke.

[e] One test value.

Effects of Cerium and Manganese Contents:

The effects of manganese were determined in the range 0.0 to 1.9 per cent in a 2 per cent cerium base and in a 4 per cent cerium base. The tensile properties at room temperature and 600 F. and minimum creep rate and total deformation at the end of 120 hr. at 600 F. are shown in Table II. The following conclusions may be drawn from these data:

1. The manganese content in the range 0 to 1.9 per cent does not have an appreciable effect on the room-temperature tensile strength, yield strength, or per cent elongation of the extrusion containing 2 or 4 per cent cerium.

2. The tensile strength and yield strength at 600 F. increase appreciably with manganese content up to approximately 1 per cent. No advantage is obtained by increasing the manganese above this amount.

3. The creep resistance is poor in the manganese-free alloys in the HTAS (solution heat treated at 1040 F. for 24 hr., air cooled, aged 16 hr. at 400 F., stabilized 24 hr. at 650 F. and air cooled) condition. The creep resistance is improved markedly by the addition of the manganese, but no further improvements appear to be obtained by additions greater than approximately 0.6 to 0.8 per cent manganese, or possibly even lower amounts.

4. When the manganese is approximately 1 per cent, the 4 per cent cerium base has a higher ultimate tensile strength at 600 F. than when the cerium content is 2 per cent. However, the higher cerium base does not produce any improvement in the room-temperature tensile properties or in the yield strength at 600 F. But the higher cerium content, apparently, has an adverse effect on the creep resistance at 600 F. under a 2000 psi. load.

The effects of cerium content in a base containing 1.5 to 1.9 per cent manganese are shown by the data in Table III. This table also shows the effect of annealing magnesium-cerium-manganese alloys and the relative merits of air cooling *versus* quenching from the heat-treating temperature.

The following conclusions may be drawn from the data in Table III.

1. In the 1.6 to 1.8 per cent manganese base, the addition of about 1.4 per cent cerium slightly improves both the ultimate strength and yield strength at room temperature, though the data are slightly variable. No appreciable additional advantage is obtained by increasing the cerium content above approximately 1.4 per cent. The cerium content in the range 0 to 6 per cent does not appear to have an appreciable effect on the room-temperature elongation of the alloys in the HTA condition.

2. Increasing the cerium content increases the ultimate tensile strength and yield strength at 600 F. This improvement apparently continues with cerium content up to at least 6 per cent, but the improvement obtained with additions greater than 1.4 per cent cerium is not very marked.

3. The cerium content does not have a very marked effect on the creep resistance of the manganese-containing alloys in the HTAS condition at 600 F. There does, however, appear to be a minimum in the creep rate and in the total deformation at the end of 120 hr. when the cerium content is in the range of 1.4 to 2.4 per cent. It will be shown later that the creep rates of the magnesium-cerium wrought alloys of the EM62, EM42, and EM22 types, at 600 F. under a 2000 psi. load, are quite variable and that this variability ap-

pears to increase with the cerium content.

4. Annealing these manganese-containing alloys has a very harmful effect upon resistance to creep at 600 F. whether they contain cerium or not.

TABLE IV.—TYPES OF ALLOYS INVESTIGATED.

Base[a]	Addition	Number of Heats
Mg-Ce	None	4
EM62 (6 per cent Ce, 1.8 per cent Mn)	None	16
EM42 (4 per cent Ce, 1.8 per cent Mn)	None	6
EM22 (2 per cent Ce, 1.8 per cent Mn)	None	5
AZ91 (9 per cent Al, 0.5 per cent Zn)	....	1
Mg—2 per cent Ce	0 to 1.9 per cent Mn	7
Mg—4 per cent Ce	0 to 1.7 per cent Mn	5
Mg—1.8 per cent Mn	0 to 6 per cent Ce	8
Mg—3-4 per cent Ce	Zr, Zr + Zn	6
Mg—6 per cent Ce, 1.8 per cent Mn	B, Ba, Bi, Cd, Cu, In, Pb, Sb, Sn, Si, Te, Tl, Zn, Zr, Be, Li, Ca, Al, Ag, Na, Sr, W, Mo, K, Ni, Fe, Ge, V, Ti, Co, Cr, P, As, Ch, Ta, In	103
Mg—2 per cent Ce, 0.7 to 1.9 per cent Mn	Al, Cd, Ni, Sr, Te	11
Mg—1.7 per cent Mn + 0.2 per cent Ni	W	1
Mg—2 per cent Ce, 0.7 to 1.9 per cent Mn, 0.2 per cent Ni	Ag, Al, Cd, Co, Mo, Sr, Te, Ti, W, Cd + W, Sr+Ag, W+ Ag, Zr + Ag, Zr + Be + Ag	19
Mg—2 per cent Ce, 1.7 per cent Mn, 0.2 per cent Ni	....	3

[a] Mg—Magnesium
Ce—Cerium
Mn—Manganese
Al—Aluminum
Zn—Zinc
Ni—Nickel.

5. Quenching from the solution-heat-treating temperature has a variable effect on the creep resistance at 600 F. Although the results are not consistent, quenching from the solution-heat-treating temperature appears to have a slightly beneficial effect on the resistance to creep. At 0 per cent cerium, this effect is very marked, and the highest resistance to creep at 600 F. was obtained in the cerium-free, 1.8 per cent manganese alloy, quenched from the solution-heat-treating temperature of 1040 F.

6. Apparently, the presence of some cerium in the alloy makes it possible to use air cooling from the solution-heat-treating temperature and still obtain good resistance to creep at 600 F.

On the basis of these data, it is quite evident that both cerium and manganese play important rôles in producing good high-temperature properties at 600 F. It also appears that a very satisfactory base composition is one which contains approximately 1.5 per cent manganese and 2.0 per cent cerium. Most of the alloy development was carried out on this base, although some of the early work was conducted on a base containing 6 per cent cerium and 1.8 per cent manganese.

Effects of Various Additions to the EM62 and EM21 Bases:

Approximately 195 heats were prepared of the types listed in Table IV. Of these heats, approximately 27 were the EM62, EM42, or EM22 types, and 24 were employed to establish the effects of manganese and cerium content. Most of the other heats prepared were composed of a base of 6 per cent cerium and 1.8 per cent manganese, or 2 per cent cerium and about 1.5 per cent manganese.

Tensile properties at room temperature and 600 F. were obtained on all of these compositions, and most of them were also subjected to the standard creep test at 600 F. under 2000 psi. load.

Figure 5 shows the total creep in 120 hr. and the minimum creep rate obtained on a number of experimental alloys containing 6 per cent cerium,

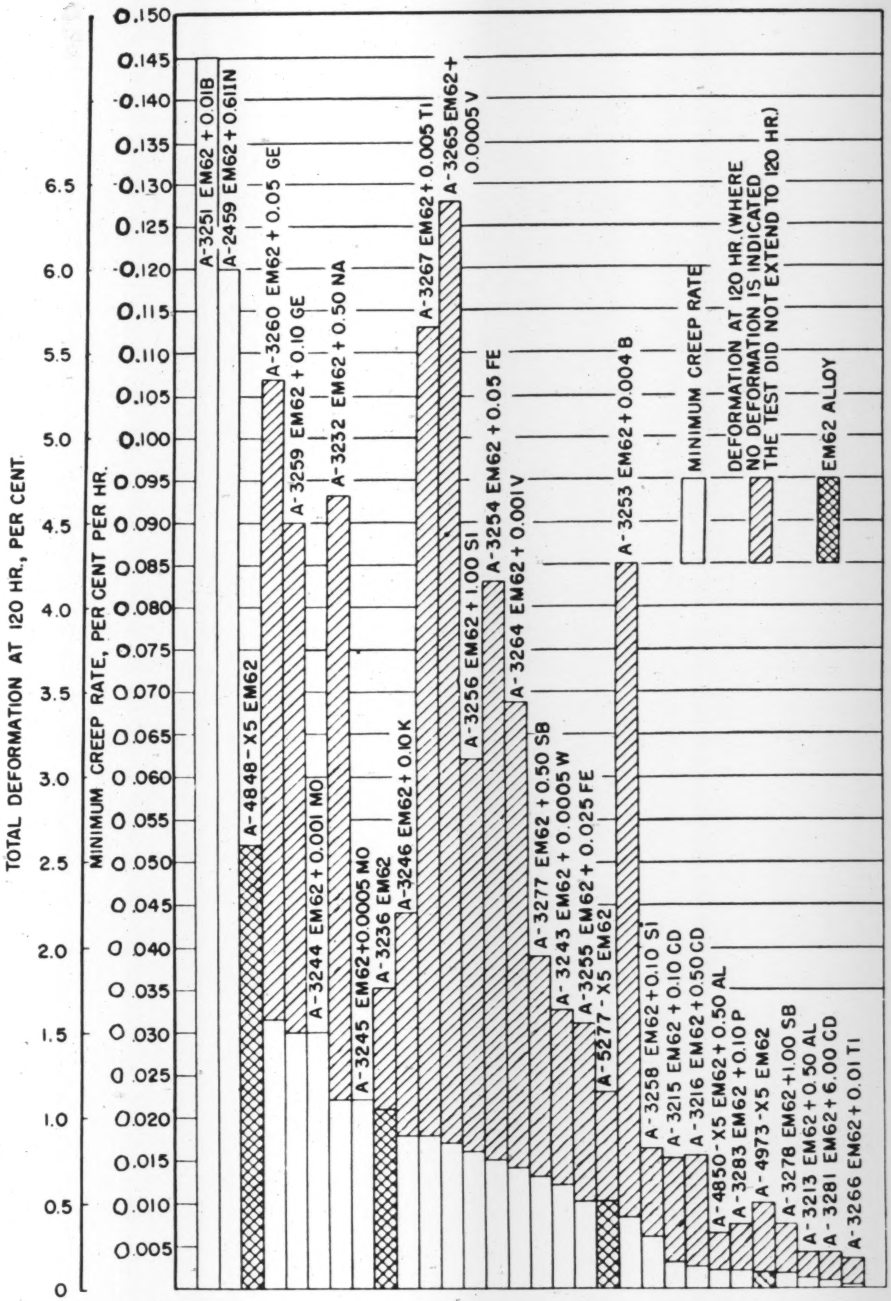


FIG. 5.—The Effects of Small Alloy Additions on the Total Deformation and Minimum Creep Rate of Extruded Magnesium Alloys Containing 6 per cent Cerium and 1.7 per cent Manganese. Tested at 600 F.—2000 psi. Load. (Note Scatter in Values for EM62 Composition.)

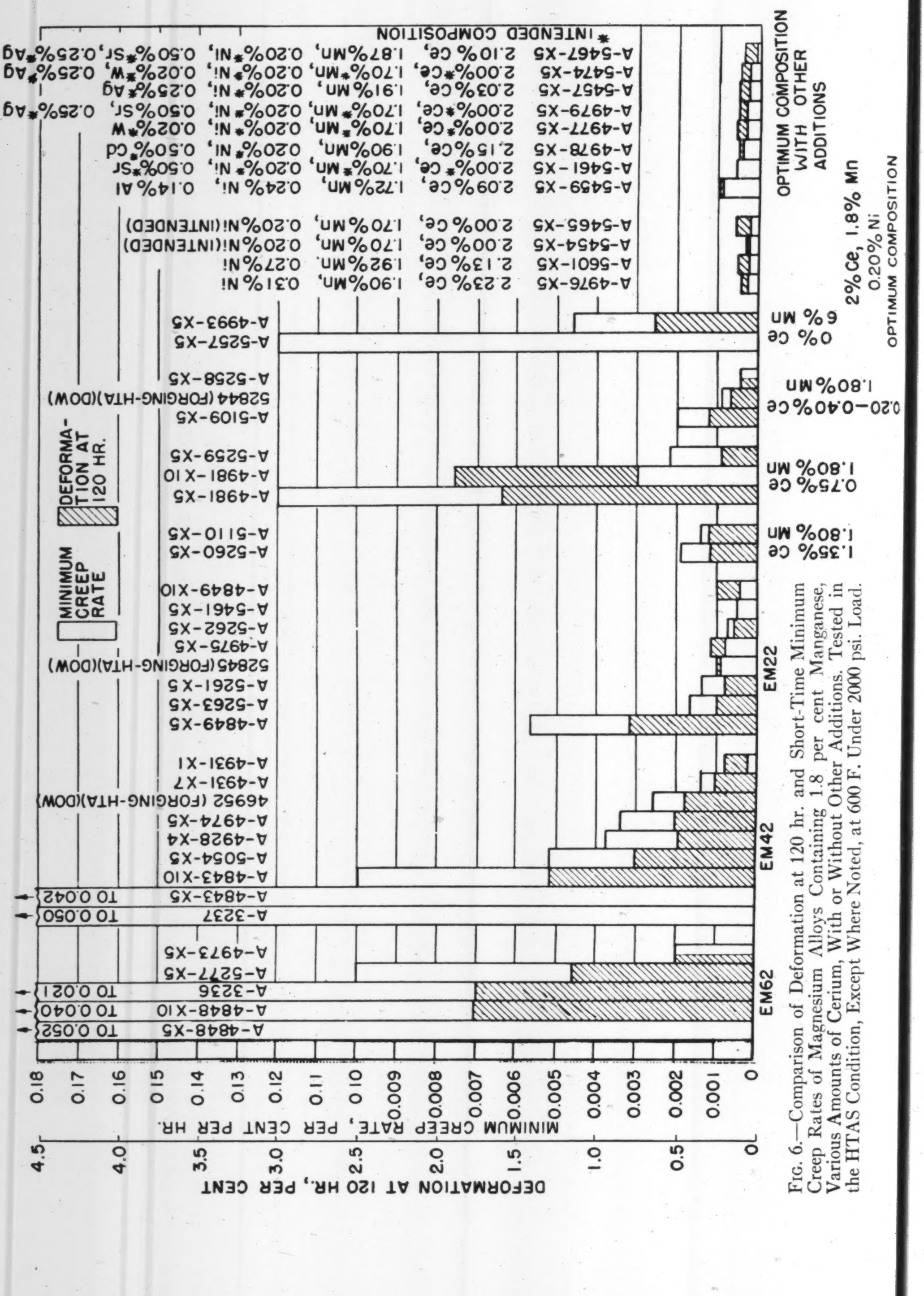


FIG. 6.—Comparison of Deformation at 120 hr. and Short-Time Minimum Creep Rates of Magnesium Alloys Containing 1.8 per cent Manganese, Various Amounts of Cerium, With or Without Other Additions. Tested in the HTAS Condition, Except Where Noted, at 600 F. Under 2000 psi. Load.

1.8 per cent manganese, and other additions. Data are also represented on three heats of the EM62 base. Considerable variation in the creep properties of this base is noted. Several additions appear to have a beneficial effect on the resistance to creep, though, in view of the variability of the test results, the data are not conclusive. Figure 6 shows similar data on a number of EM62, EM42, and EM22 heats as well as several low-cerium alloys and alloys containing 2 per cent cerium, 1.8 per cent manganese, 0.2 per cent nickel, with and without small additions of other elements. This figure clearly shows the variability in the resistance to creep of the EM62, EM42, and EM22 alloys. The resistance to creep appears to increase and the variability in results to decrease as the cerium content is lowered to 2 per cent.

Because nickel was found to be a useful addition in the cast alloys, as described in another paper,[4] it was also tried in the wrought alloys containing 2 per cent cerium and 1.8 per cent manganese. It was found that this addition of 0.2 per cent nickel increased the ultimate tensile and yield strengths at 600 F. by about 2000 psi. each and, fortunately, at the same time, improved the resistance to creep. Figure 6 shows the consistently low minimum creep rates and total deformation in 120 hr. obtained on four different compositions containing 2 per cent cerium, 1.7 to 1.9 per cent manganese, and 0.2 per cent

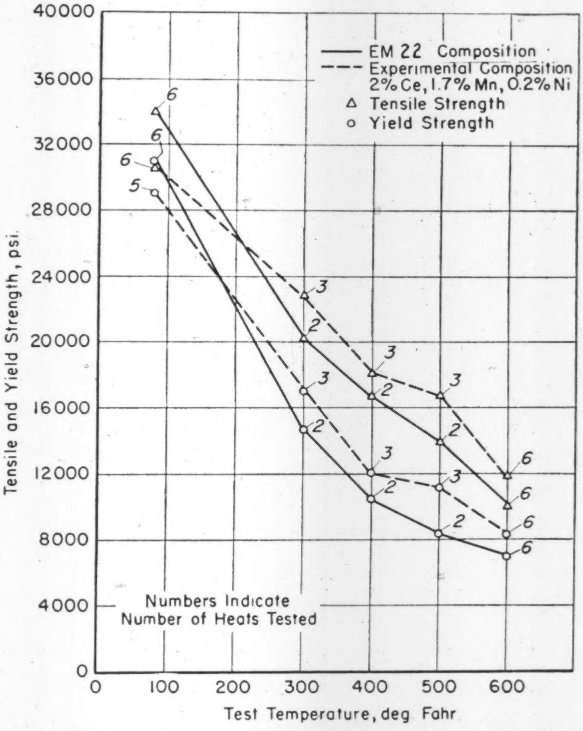


FIG. 7.—The Effect of Temperature on the Tensile Properties of Extruded EM 22 Alloy and Experimental Alloy Containing Nickel. Tested in the HTA Condition at Room Temperature and HTAS Condition at Elevated Temperatures.

nickel. None of the small additions of other elements to this 2 per cent cerium-1.8 per cent manganese-0.2 per cent nickel base appeared to produce any additional improvement in the resistance to creep. Experience has shown that, when the creep rates are of the order of 0.0005, it becomes difficult to attain still lower creep rates. Consequently, at slightly higher loads and creep rates, it is easier to measure any improvements produced by adjusting the composition. However, the results obtained on the 195 different heats show that the alloy of optimum composition appears to be as follows:

2 per cent cerium
1.2 to 1.8 per cent manganese
0.2 per cent nickel

The compositional limits of this alloy were not established. Because this alloy appeared to have excellent high-temperature properties, considerable data were obtained on it.

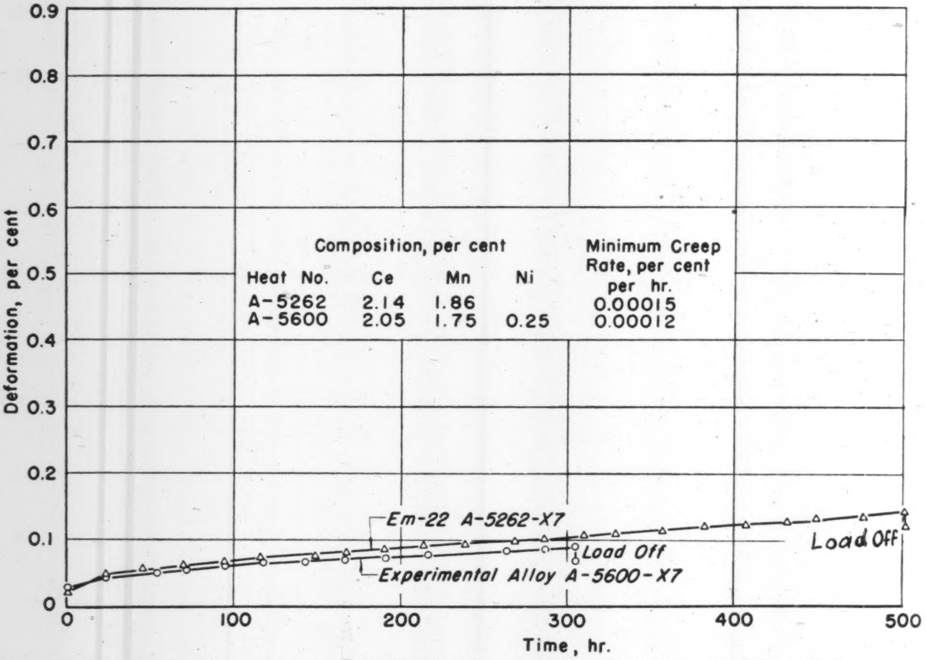


FIG. 8.—Time-Deformation Curves of EM 22 Alloy and the Experimental Alloy of Optimum Composition in the HTAS Condition. Tested at 600 F.—1300 psi. Load.

PROPERTIES OF ALLOYS

Figure 7 shows the effects of temperature on the tensile strength and yield strength of the EM22 composition and the experimental alloy of optimum composition. The improved tensile properties obtained on the alloy of optimum composition throughout the range of 300 to 600 F. are apparent.

Figures 8, 9, and 10 show time-deformation curves for EM22 and the alloy of optimum composition at 600 F. using various loads. Figure 11 shows the design curves constructed from the time-deformation data on the two alloys

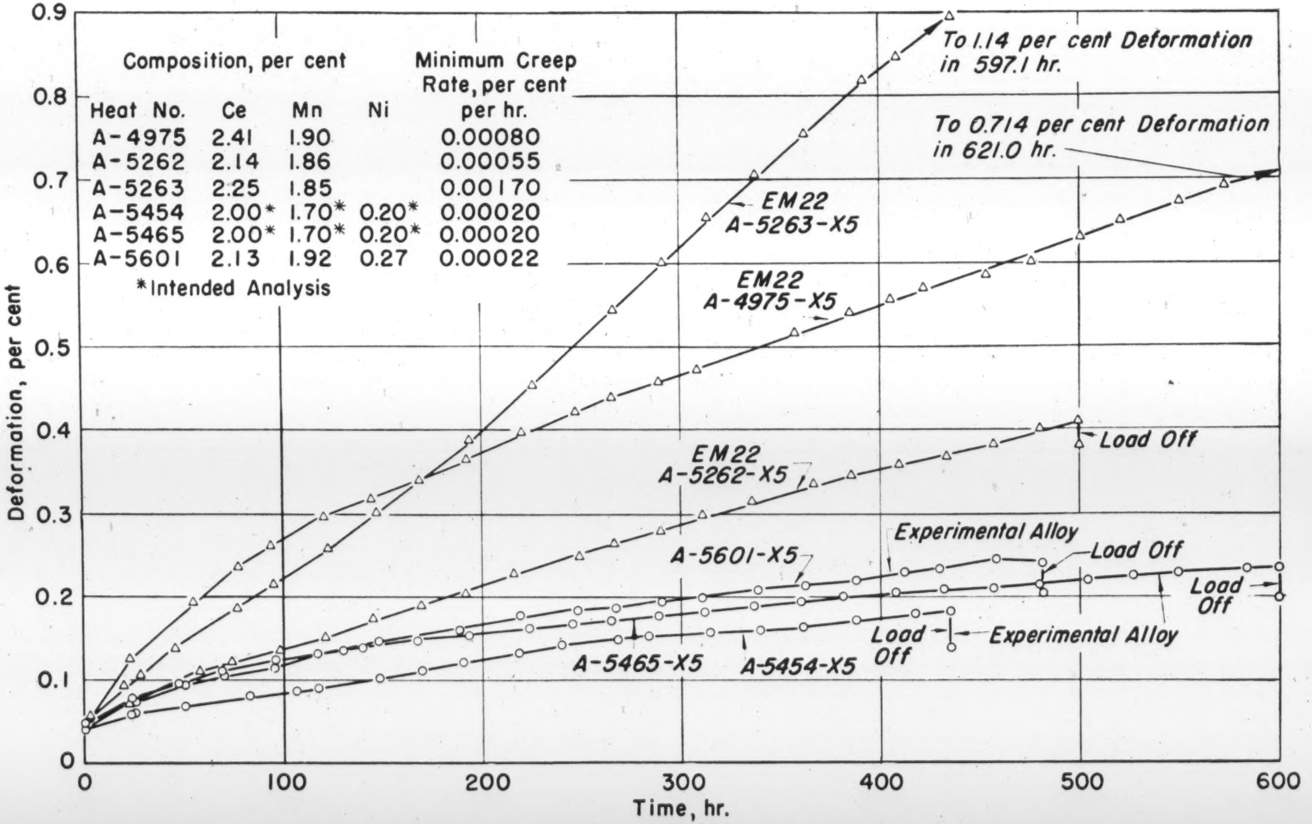


FIG. 9.—Time-Deformation Curves of Three Heats of EM 22 Alloy and Three Heats of Experimental Alloy of Optimum Composition. Tested in the HTAS Condition at 600 F.—2000 psi. Load.

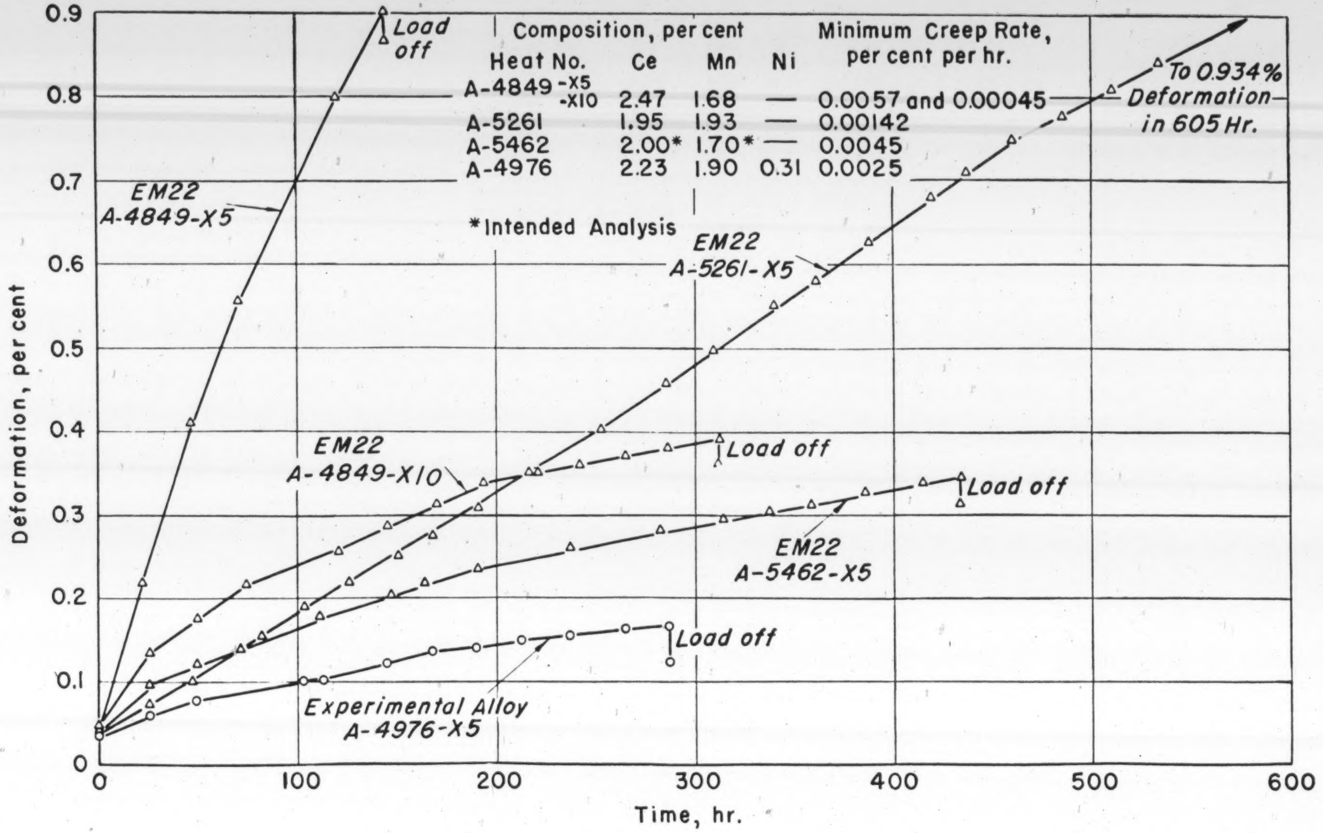


FIG. 10.—Time-Deformation Curves of Three Heats of EM 22 Alloy and One Heat of Experimental Alloy of Optimum Composition. Tested in the HTAS Condition at 600 F.—2000 psi. Load.

at 600 F. The improved resistance to creep of the new alloy and its greater consistency are clearly shown by the creep data.

Figure 12 shows microstructures of the alloy of optimum composition after heat treating at 1040 F. and 1000 F. It will be noted from the data in the captions that very much better creep resistance is obtained by using the higher heat-treating temperature. At this temperature, there appears to be a considerable increase in the amount of magnesium-cerium constituents dissolved, some grain coarsening, and a small amount of grain-boundary melting. No difficulties were noted when heat treating at the temperature of 1040 F. using the usual low percentage of SO_2 in the atmosphere.

The magnesium-cerium wrought alloys, like the magnesium-cerium casting alloys, are subject to some oxidation when exposed for a prolonged period at 600 F. and higher. It has been found that this oxidation increases slightly with the cerium content. Alloys which are to be subjected to such temperatures for prolonged periods probably should be protected. The fluosilicic acid treatment, described in another paper,[4] was not tried on the wrought alloys, but in all probability it would be effective as a means of reducing oxidation.

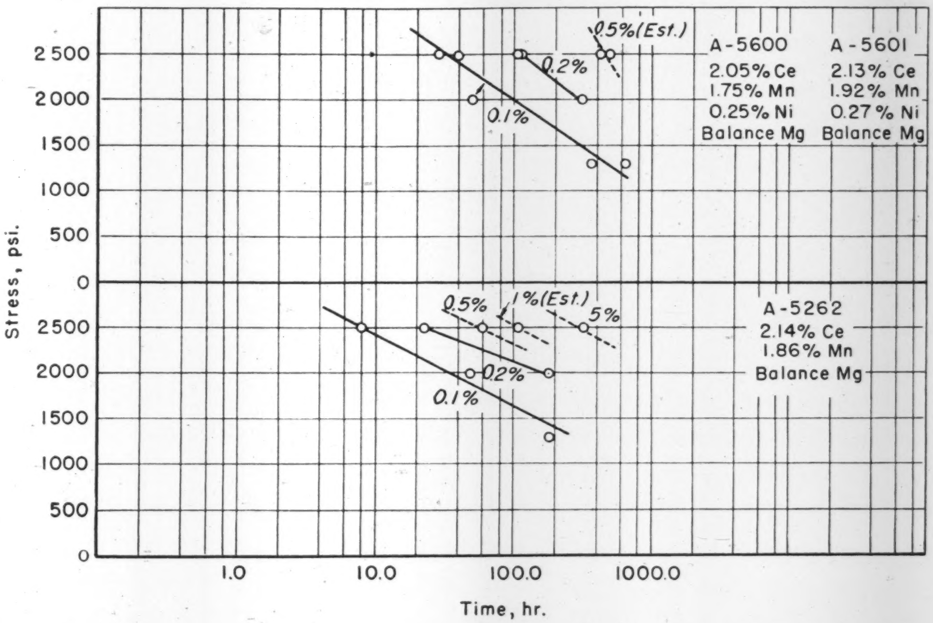
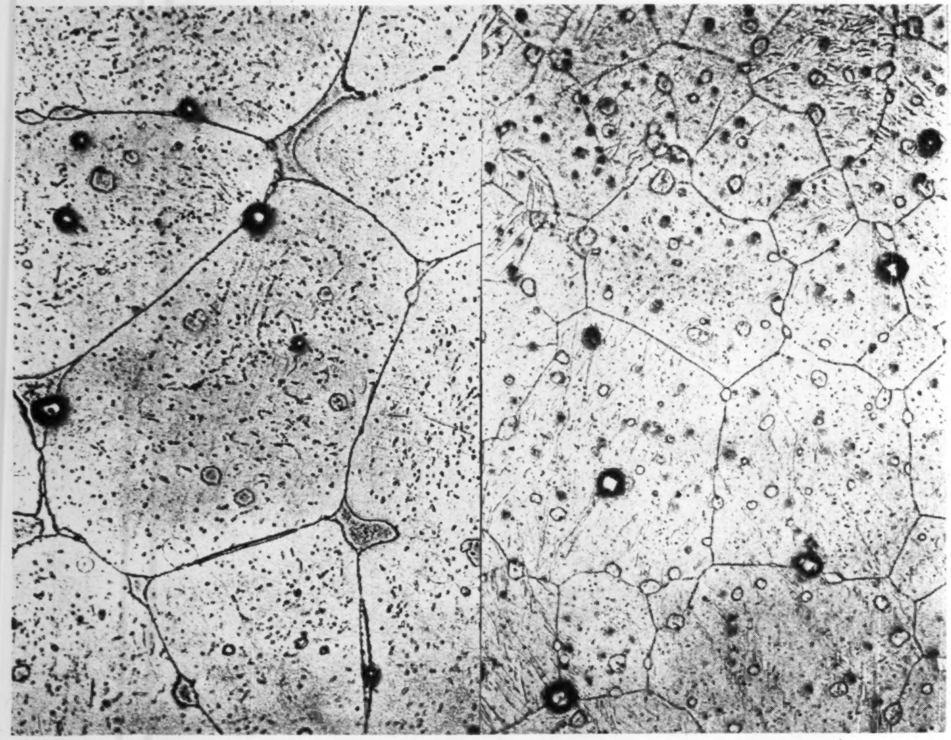


FIG. 11.—Comparison of Some Design Data on Extruded EM 22 Alloy and Two Heats of the Experimental Alloy of Optimum Composition. Tested in the HTAS Condition of 600 F.

A few forging tests were conducted on the EM42 and experimental alloy of optimum composition. It was found that the round extrusion ingots prepared by the water-cooling process, described previously in this paper were not suitable for forging tests. Consequently, the alloys were chill-cast into the form of ingots 5 by 5 by 8 in. high. The ingot mold employed was of gray iron fitted with a copper bottom, and with a refractory hot top to provide adequate feeding during solidification. Experimental forging tests were performed on five heats. Although the tests were too limited to permit definite conclusions, the alloy of optimum composition appears to forge about as well as the EM42 alloy. The optimum forging temperature is about 950 F., as shown by these preliminary tests.



(*a*) Heat Treated at 1040 F. (HTAS)
Minimum Creep Rate, per cent per hr.—0.00025
Deformation in 120 hr., per cent—0.110
Duration of Test, hr.—287
Total Deformation, per cent,—0.167

(*b*) Heat Treated at 1000 F. (HTAS)
Minimum Creep Rate, per cent per hr.—0.0015
Deformation in 120 hr., per cent—0.262
Duration of Test, hr.—284.3
Total Deformation, per cent—0.635

FIG. 12.—Microstructure of Optimum Composition Alloy (2.23 per cent Cerium, 1.90 per cent Manganese, 0.31 per cent Nickel)(×250).

Creep rate at 600 F., 2000 psi.

EFFECTS OF FABRICATING PROCEDURE ON THE PROPERTIES OF THE ALLOYS

Several of the alloys were tested in the cast, extruded, and rolled conditions. It was found that the method of fabrication had a considerable effect upon the properties at room temperature and at 600 F. Table V shows the tensile and creep properties of several alloys fabricated by different methods. The specimens were tested at room temperature in the HTA condition, whereas those tested at 600 F. were in the HTAS condition.

As would be expected, the castings have the lowest tensile properties at room temperature. The tensile properties of the sheet are second best, while those obtained on extrusions are the best at room temperature. At 600 F.,

TABLE V.—EFFECT OF METHOD OF FABRICATION ON THE TENSILE AND CREEP PROPERTIES OF CERIUM-MANGANESE-MAGNESIUM ALLOYS.

Heat	Analyses, per cent			Method of Fabrication	Heat Treatment[b]	Test Temperature, deg. Fahr.	Tensile Properties[a]				Creep Properties, 2000 psi. Load						Remarks
	Cerium	Manganese	Others				Yield Strength, 0.20 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Duration, hr.	Initial Deformation, per cent	Minimum Creep Rate, per cent per hr.	Deformation at End of 120 hr., per cent	Total Deformation, per cent	Contraction on Release of Load, per cent	
2 per cent Cerium																	
A4849	2.47	1.68		Extrusion	HTA	70	27 900	30 500	9.0	9.2							
					HTAS	600	6 500	10 500	100.0	79.0	144.5	0.045	0.0057	0.800	0.902	0.036	
					HTAS	600					313.0	0.047	0.00045	0.255	0.393	0.032	
				Sand cast	HTA	70	5 060	8 750	35.0	45.9							
				¼-in. rolled plate	HTA	70	13 550	24 000	4.1								Flaw
A4975	2.41	1.90		Extrusion	HTA	70	27 250	33 000	7.2	10.6							
					HTAS	600	7 400	9 950	116.0	83.2	621	0.049	0.0008	0.294	0.714	0.027	Grain size—0.003 in.
					HTQAS	600					601	0.050	0.0035	0.500	3.04	.....	Water quenched.
					ANN.	600					51	0.055	0.175	.....	11.04	0.040	
				Sand cast	HTAS	600	6 550	9 075	72.0	55.3	307	0.041	0.00015	0.080	0.133	0.048	Grain size—0.05 to 0.06 in.
					HTAS	600					304	0.044	0.00007	0.132	0.150[c]		
				Sand cast	HTAS	600					288	0.041	0.00030	0.122	0.178	0.035	Grain size—0.02 in.
					HTAS	600					312	0.046	0.00012	0.072	0.104	0.039	
				0.060-in. rolled sheet	HTA	70	15 250	26 550	9.8								Parallel to the direction of rolling.
					HTAS	600		8 325	51.5		150	0.024	0.0055	0.750	0.982	0.026	
					HTAS	600					150	0.029	0.0165	3.50	4.95	.....	
				0.060-in. rolled sheet	HTA	70	14 125	25 700	8.0								Transvere to direction of rolling.
					HTAS	600		7 125	40.0		211	0.023	0.0065	0.812	1.89	0.010	
					HTAS	600					208.7	0.023	0.012	2.27	7.08	0.020	
A5462	2.00[d]	1.70[d]		Extrusion	HTA	70	28 250	31 700	6.0	7.2							
					HTAS	600	6 700	9 300	85.0	54.2	434	0.042	0.00045	0.186	0.349	0.034	
				0.080-in. rolled sheet	HTA	70	15 000	26 650	10.5								
					HTAS	600		7 825	80.0								
A4976	2.23	1.90	0.31 Nickel	Extrusion	HTA	70	26 200	32 250	7.0	7.3							Solution Heat Treated—1000 F.
					HTA	70	30 700	32 250	1.5	1.1							
					HTAS	600	8 700[e]	12 500[e]	132.5[e]	84.0[e]	284.3		0.0015	0.262	0.635		Solution Heat Treated at 1000 F.
					HTAS	600	9 375	12 700	84.2	67.5	287	0.041	0.00025	0.110	0.167	0.043	
				Sand cast	HTAS	600	6 500	9 400	3.0 to 54.0	2.8 to 48.4	336	0.039	0.00025	0.083	0.182	0.048	
					HTAS	600					312	0.041	0.00032	0.095	0.160	0.044	

[Continued on pages 984 and 985]

TABLE V.—*Continued*

Heat	Analyses, per cent			Method of Fabrication	Heat Treatment[b]	Test Temperature, deg. Fahr.	Tensile Properties[a]				Creep Properties, 2000 psi. Load						Remarks
	Cerium	Manganese	Others				Yield Strength, 0.20 per cent Offset, psi.	Tensile Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Duration, hr.	Initial Deformation, per cent	Minimum Creep Rate, per cent per hr.	Deformation at End of 120 hr., per cent	Total Deformation, per cent	Contract on on Release of Load, per cent	
2 per cent Cerium—*Continued*																	
A5600	2.05	1.75	0.25 Nickel	Extrusion	HTA	70		28 425	1.0	1.0							
					HTA	300	18 400	22 550	3.7	4.0							
					HTAS[f]	400	14 125	19 500	10.2	14.1							
					HTAS[g]	500	11 750	17 300	20.0	18.5							
					HTAS	600	8 200	11 500	51.5	39.2	509	0.026	0.00012	0.037	0.077	0.020	1300 psi. stress.
					HTAS	600					305	0.028	0.00012	0.063	0.088	0.023	1300 psi. stress.
					HTAS	600					312	0.049	0.0009	0.213	0.403	0.050	2500 psi. stress.
				Sand cast	HTA	70		10 000	0.8	0.8							
					HTAS	500	6 750	10 100	17.0 to 60.0	20.6 to 43.8	232	0.042	0.00048	0.125	0.187	0.023	
				0.070-in. rolled sheet	HTA	70	10 425	17 775	3.5								Damaged in heat treatment.
					HTAS	600		7 900	66.5								
A5601	2.13	1.92	0.27 Nickel	Extrusion	HTA	70	27 000	29 250	1.7	1.4							
					HTA	300	16 700	22 375	9.2	9.0							
					HTAS[f]	400	11 000	16 250	18.0	23.7							
					HTAS[g]	500	10 200	14 950	27.0	41.2							
					HTAS	600	7 325	10 950	92.7	66.2	482	0.042	0.00022	0.130	0.242	0.044	
					HTAS	600					338	0.055	0.00088	0.205	0.393	0.042	2500 psi. stress.
				Sand cast	HTA	70		9 000	0.8	0.6							
					HTAS	600	6 425	9 450	5.0 to 45.0	3.9 to 48.2	233	0.038	0.00005	0.058	0.065	0.033	
A4977	2.00[d]	1.70[d]	0.20 Nickel[d] 0.02 Wolfram	Extrusion	HTA	70	28 850	30 000	1.0[e]	1.1[e]							
					HTAS	600	9 900	13 400	62.5	53.6	312	0.049	0.00030	0.135	0.200	0.031	
				Sand cast	HTA	70		13 125	1.0	1.0							
					HTAS	600	6 850	10 350	62.0	59.0							
A5464	2.00[d]	1.70[d]	0.20 Nickel 0.50 Zirconium 0.01 Beryllium[d]	Extrusion	HTA	70	28 000	30 900	2.5	2.8							
					HTAS	600	8 850	12 100	93.5	72.0							
				0.080-in. rolled sheet	HTA	70	14 900	26 650	12.5								
					HTAS	600		8 350	38.0 to 85.0								

4 per cent Cerium																
A4846	4.50	0.62		Extrusion	HTA	70	26 175	30 850	6.5	9.2[e]						
					HTAS	600	6 150	10 400	126.0	92.7	47	0.043	0.0905	.....	3.88	0.040
					HTAS	600					143	0.045	0.030	4.67	5.64	0.040
				¼-in. rolled plate	HTA	70	18 400	27 000	6.1							
A4845	4.50	1.03		Extrusion	HTA	70	25 250	30 700	5.5							
					HTAS	600	6 575	10 675	125.0	90.3	93	0.041	0.0595	.....	5.04	0.040
					HTAS	600					142	0.038	0.0230	4.43	5.65	0.050
A4845	4.50	1.03		¼-in. rolled plate	HTA	70	18 150	26 950	5.7							
A4844	4.39	1.38		Extrusion	HTA	70	24 850	30 250	3.5	4.9						
					HTAS	600	6 700	10 800	129.0	86.1	149	0.045	0.0195	2.85	3.18	0.040
					HTAS	600					143	0.043	0.0160	5.00	6.08	0.050
				¼-in. rolled plate	HTA	70	20 500	27 200	4.1							
6 per cent Cerium																
A4848	6.27	1.60		Extrusion	HTA	70	31 400	33 100	3.5	3.9						
					HTAS	600	7 400	12 150	121.0	85.5	114	0.041	0.0520	.....	4.92	0.050
					HTAS	600					194	0.043	0.014	1.76	2.68	0.040
				Sand cast	HTAS	600	6 530[e]	11 075	35.0	37.0						
A4850	6.00[d]	1.70[d]	0.50A1[d]	Extrusion	HTA	70	27 550	29 875	4.0	1.9						
					HTAS	600	6 400	11 600	97.5	80.8	145	0.047	0.0023	0.340	0.412	0.040
				Sand cast	HTAS	600	5 500[e]	11 000[e]	27.0[e]	26.1[e]						
				¼-in. rolled plate	HTA	70	18 825	23 750	3.3							

[a] Average of 2 tests.

[b] HTA—Solution heat treated 24 hr. at 1040 F., air cooled, and aged 16 hr. at 400 F.
HTAS—Solution heat treated and aged as above, followed by a 24-hr. at 650 F. stabilization treatment.
HTQAS—Quenched in cold water from the solution heat-treating temperature employed above. Aged for 16 hr. at 400 F. followed by stabilizing 24 hr. at 65. F.
ANN.—Furnace cooled from the solution heat-treating temperature of 1040 F.

[c] Estimated value.

[d] Intended analyses.

[e] One test only.

[f] Stabilized 24 hr. at 500 F.

[g] Stabilized 24 hr. at 575 F.

the tensile properties of the extrusions are equivalent to or slightly better than those of the castings, but the tensile properties of the sheet are again somewhat inferior to those of extrusions. The average creep values obtained on 13 extruded specimens, 7 cast specimens, and 4 sheet specimens of the 2 per cent cerium, 1.7 per cent manganese alloy, with or without 0.2 per cent nickel, were as follows:

Property	Castings	Extrusions	Sheet
Minimum creep rate (2000 psi., 600 F.), per cent per hr.	0.0002	0.0012	0.010
Total deformation, per cent in 120 hr.	0.096	0.244	1.844

The average minimum creep rate of the castings in the standard test is lowest, that of the extrusion six times higher, and that of 0.060-in. sheet about fifty times higher. The greater resistance of the castings to creep is typical of other light alloys at 600 F. These effects of fabricating techniques are a function of differences in structure of the alloy and differences in geometry of the test specimens. The test specimens for the castings and extrusions were similar.

RELATIONSHIP OF COMPOSITION AND STRUCTURE TO THE CREEP PROPERTIES

The data obtained to date indicate definitely that it is mainly the manganese and, to some extent, the cerium in solution at the heat-treating temperature which have a beneficial effect on the creep properties of these alloys. Cerium is primarily responsible for good tensile properties at 600 F., but it is the manganese which imparts good creep resistance. However, it should be noted that the employment of both cerium and manganese in an alloy makes it possible to obtain good creep resistance without rapid quenching from the solution-heat-treating temperature. Increasing the cerium content to an amount greater than that which is soluble appears to have a harmful effect on the creep resistance of the extruded rod, though this did not appear to be the case with castings. While these factors certainly are the underlying ones determining the creep properties of the magnesium-cerium-manganese wrought

TABLE VI.—COMPARISON OF THE AVERAGE PROPERTIES OF THE EXPERIMENTAL ALLOY OF OPTIMUM COMPOSITION WITH OTHER WROUGHT ALLOYS.

Alloy	BMI Alloy	EM22	32ST[a]	18S[a]
Form	Extrusion	Extrusion	....	....
Condition	HTAS	HTAS	Stabilized	Stabilized
Tensile strength, psi.:				
Room temperature	30 500	34 000	54 000	63 000
400 F.	18 000	16 500	16 000	20 000
600 F.	12 000	10 000	6 000	5 000
Tensile yield strength, 0.2 per cent offset, psi.:				
Room temperature	28 000	30 500	46 000	47 000
400 F.	12 000	10 500	11 000	15 000
600 F.	8 200	7 000	3 500	3 000
Elongation in 2 in. per cent:				
Room temperature	4.0	5.0	8.0	17.0
400 F.	26.0	26.0	30.0	26.0
600 F.	100.0	90.0	60.0	65.0
C_L = load, to produce 0.5 per cent deformation in 100 hr. at 600 F., psi.	2 800	2 300	2 300	3 000
$C_L \div$ density	1 550	1 270	860	1 070
Creep rate, 2500 psi. load at 600 F., per cent per hr.	0.001	0.01	0.1	0.001
Total deformation in 120 hr. at 600 F., 2500 psi. load, per cent	0.205	1.0	......	......

[a] Data from Air Force Project RAND Report, R104, "The Effects of Temperature on the Properties of Aluminum Alloys" by C. M. Craighead, L. W. Eastwood, and C. H. Lorig.

alloys, there are many variations from heat to heat and even from one test specimen to another from the same heat. These variations are not easily understood. It does not follow that any alloy addition which is soluble at the heat-treating temperature will be beneficial. Most such additions are either without benefit or harmful to the creep resistance.

It is interesting to note that specimens cut from sheet, parallel to and transverse to the direction of rolling, had substantially the same resistance to creep at 600 F. Data were not obtained on the degrees of preferred orientation in the two directions. It is assumed, however, that there would be an appreciable difference in the two directions, a result of difference in angular scatter of hexagonal basal planes. This difference, if it existed at all, apparently did not produce any difference in creep resistance.

Further studies are required before there is a comprehensive understanding of the relationship of composition and structure to the creep properties of the alloy.

SUMMARY

A systematic study has been made of about 195 heats of magnesium-cerium wrought alloys by making tension tests at room temperature and at 600 F. and creep tests at 600 F. Manganese up to 1 per cent or more and cerium up to 2 per cent are very beneficial. The alloy of optimum composition appears to be as follows:

Cerium (rare-earths)—2 per cent
Manganese—1 to 1.5 per cent
Nickel—0.2 per cent

The compositional limits were not established. The comparison of the properties of this alloy with those of EM22, 18S, and 22S alloys is shown in Table VI. It is quite evident that the new alloy is appreciably superior to EM22 or the two high-temperature aluminum-base alloys at 600 F.

Acknowledgments:

The authors gratefully acknowledge the support of the National Advisory Committee for Aeronautics, which made this investigation possible, and who granted permission for its publication, the guidance and encouragement of C. H. Lorig, the assistance of H. C. Cross, and Ward Simmons of Battelle, who conducted the creep tests, and the careful efforts of Ray Kaiser who did much of the laboratory work.

DISCUSSION

Mr. J. W. Meier.[1]—I should like to ask the following questions:

(*a*) Did the authors find any tendency towards segregations of the heavy metals in the cast extrusion billets?

(*b*) Is there any effect of the billet-heating time on the properties of the extrusions?

(*c*) Have the authors any information on the "workability" and the welding characteristics of the proposed alloy, and on the effect of working or welding on the elevated temperature properties?

Mr. L. W. Eastwood (*author*).—We have always had very satisfactory results with ingots prepared by chill casting as described in the paper. There was no evidence of difficulties with segregation. On the other hand, no study was undertaken to check their uniformity in composition.

No study was made of the effects of soaking time prior to extrusion and no information was obtained on weldability. The hot-working characteristics probably will be similar to other magnesium-cerium alloys. Alloys of this type are much harder at forging temperatures than the common magnesium-base alloys.

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MAGNESIUM-CERIUM CAST ALLOYS FOR ELEVATED-TEMPERATURE SERVICE*[1]

By K. Grube[2] and L. W. Eastwood[2]

Synopsis

About 350 heats of magnesium-cerium alloys, most of which contained other elements, have been prepared and studied. The evaluation of the compositions was based on the following tests:

1. Tension tests at room temperature.
2. Tension tests at 600 F.
3. Creep tests at 600 F., using a 2000 psi. and a 2500 psi. load.
4. Macro and microexaminations.

Six per cent cerium (rare earths) was selected for study as a compromise between cheaper and lower density alloys of lower cerium content, on the one hand, and higher strength alloys of still higher cerium content, on the other. Manganese was also employed in most of the alloys because of its beneficial effects on tensile properties and creep resistance at 600 F. The alloy of optimum composition tentatively selected is as follows:

Cerium (rare earths), per cent	6
Manganese, per cent	0.8
Nickel, per cent	0.2
Tungsten, per cent	0.02

The designer and metallurgist are constantly seeking structural materials having improved strength-weight properties at the temperature of the part during service. Because of their low density and high strength at normal or slightly elevated temperatures, magnesium castings have been used in aircraft motors for a number of years. For the most part, they were employed only for such parts of the motor as operated at moderate temperatures because, as is well known, the standard magnesium-aluminum-zinc alloys such as AZ92 (9 per cent aluminum, 2 per cent zinc) and AZ63 (6 per cent aluminum, 3 per cent zinc) have relatively poor creep resistance at temperatures appreciably above 300 F. Magnesium rare-earth alloys possessing improved elevated-temperature properties were first described by J. L. Haughton and W. E. Prycherch (**2**)[3] and Adolph Beck (**1**). During the war, a study of enemy materiel showed that Germany was using magnesium rare-earth alloys for certain motor parts. Early in 1945, Battelle undertook the development of alloys of this type for the Air Technical Service Command, Wright Field, and later the work was continued under the sponsorship of the Bureau of Aeronautics. Similar developments on magnesium-rare-earth wrought alloys, sponsored by the National Advisory Committee for Aeronautics at Battelle, are described in a separate paper (**11**).

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* Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[1] The experimental work described in this paper was sponsored by the Air Technical Service Command, Wright Field, from March 1, 1945, to February 28, 1947, Contract W 33-038 ac-7202; and by the Materials Branch, Airborne Equipment Division, Bureau of Aeronautics, Navy Department, from June 1, 1947, to May 31, 1948, Contract NOa (s) 8957.

[2] Engineer and Supervisor, respectively, Battelle Memorial Inst., Columbus, Ohio.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1009.

At the time the work was started, magnesium rare-earth alloys containing 2 to 6 per cent rare earth and 0 to 1.7 per cent manganese were known. The general aim of the work described in this paper was to obtain casting alloys having better foundry characteristics than those of EM61 alloy (6 per cent rare earths, 1.5 per cent manganese) and better load-carrying capacity at 600 F., and possibly better foundry characteristics than alloys of the E6 type (6 per cent rare earths).

In the early stages of the work, considerable assistance was rendered by the Dow Chemical Co. who made available their Report No. 11694, which described the preparation of alloys and the use of their 220 flux. In addition, another report by the Dow Chemical Co., "The Properties of Binary and Polynary Magnesium Alloys Containing Cerium at Room and Elevated Temperatures," was also found to be very useful. This report was particularly valuable in that it established the proper heat-treating procedures to employ for maximum resistance to creep.

Since this work was started at Battelle, a considerable amount of information (**3, 4, 5, 6, 7**) has been published on magnesium rare-earth alloys, as shown by the bibliography at the end of this paper.

EXPERIMENTAL METHODS

With the exception of the melting technique, the experimental methods to be employed were quickly developed. The general procedure consisted of preparing the melts, pouring them into test bars, and subjecting the test bars to a heat treatment, followed by tension tests at room temperature and at 600 F., a standardized creep test at 600 F., and metallographic examination.

Melting Methods:

During the first year's work, the melting procedures employed were similar to those recommended by the Dow Chemical Co. in their Report No. 11694, using their 220 flux. Because freedom from flux inclusions could be obtained and higher cerium recoveries produced, a new method of melting was devised. By this method of melting, the steel crucible is fitted with a cover and the magnesium-cerium scrap and magnesium are melted first. During this part of the melting, dry nitrogen is introduced through the cover and blown gently over the melt, the nitrogen inlet being at right angles to the melt surface. After the first charge has been melted, the cover is removed and additional alloy scrap and magnesium melted as required. After the second addition is melted and the temperature raised to about 1400 F., the cover is removed, the cerium added along with other alloy additions, and the melt stirred until the alloy has been dissolved. The cover is then replaced, the nitrogen is again introduced over the melt, and the latter is heated to the pouring temperature. The crucible is then removed from the furnace, the cover lifted from the top, and the metal skimmed. During the pouring operation, the melt is protected with sulfur-boric acid mixtures following the usual practice.

Since a large number of different alloy additions were investigated during the course of the alloy development, considerable experience was obtained in methods of adding the alloys. Manganese was added to the melt in the form of M1 alloy (1.5 per cent manganese), and zirconium was added in the form of magnesium-0.7 per cent zirconium alloy. This zirconium alloy was prepared in advance by stirring zirconium tetrachloride into a magnesium melt. Zirconium was also added in the form of magnesium-zirconium briquets containing 50 per cent zirconium and 50 per cent magnesium. In general, the methods of adding the zirconium to the magnesium, using zirconium tetrachloride or the magnesium zirconium briquet, were equally effective. The use of the zirconium-magnesium briquet helped to some extent to avoid difficulties with flux inclusions.

Relatively insoluble additions such as columbium, molybdenum, tantalum, boron, and titanium were added to the magnesium melt prior to the addition of cerium or cerium-containing scrap. These additions were made to the melt at about 1600 F., the metal being added in a finely divided form, such as powder. Boron was added in the form of a powder of a commercial manganese-boron alloy. In general, the results obtained from these difficultly soluble elements were inconclusive.

Rare earths in the form of misch metal, referred to in this paper as "cerium," were added to the melt in the form of lumps or as the main alloy constituent in the scrap returned for remelting. Typical analyses of the misch metal have been described elsewhere (**12**).

When beryllium was used alone, it was added either in the form of beryllium chips, a beryllium-magnesium compact containing 62 per cent beryllium and 38 per cent magnesium, a flux containing 4 parts BeF_2, 1 part CaF_2, and 1 part BaF_2, or a cerium-beryllium alloy containing 98.3 per cent cerium and 1.7 per cent beryllium. Of these methods, the latter alloy addition is preferred. When beryllium and nickel or beryllium and copper were used, they were added in the form of small pieces of a nickel-beryllium alloy or a copper-beryllium alloy.

When nickel and tungsten were used together, they were added in the form of a 90 per cent nickel-10 per cent tungsten alloy which had been rolled to sheet and cut up into suitable pieces.

Alloy Additions in Form of Master Alloy.—Because the alloy of optimum composition developed by this work contains magnesium, rare earths, manganese, nickel, and tungsten, or zirconium, the problem of combining these alloy additions has received some attention. It has been found that the problem of adding these alloy elements to magnesium can be readily solved by the use of a master alloy. While these alloying elements may be introduced as single additions, the use of a master-alloy addition offers several advantages. The recovery of the various elements is more readily controlled, and nonmetallic or flux inclusions are reduced to a minimum. Further, the alloying operation is simplified by making only a single addition. A proprietary master alloy of the following approximate composition has been developed by one of the leading producers of misch metal alloys:

Cerium (misch metal), per cent	62
Magnesium, per cent	24
Manganese, per cent	12
Nickel, per cent	2
Tungsten, per cent	0.2

This and similar compositions have a melting point of approximately 1550 F. and are readily dissolved in molten magnesium at a temperature of 1550 F. The magnesium is melted under a flux or inert gas cover, and when the charge has reached the desired temperature, the master alloy addition is made. The master alloy sinks to the bottom and the bath is thoroughly stirred. The metal may be cast as soon as the master alloy has been dissolved and the melt reheated to the pouring temperature.

The composition of the master alloy can be varied over a wide range and, when desired, may include zirconium or beryllium. Such master alloys are commercially available and should be of particular value to the small jobbing foundry which is normally not equipped for extensive alloy development or chemical analysis work.

Casting:

A standard, synthetic magnesium green sand was employed, the inhibitors being sulfur, boric acid, and ethylene glycol. The test bar poured was the standard four-bar casting normally employed in magnesium foundries. The pouring temperature was 1400 to 1420 F., representative of commercial foundry operations rather than the lowest possible pouring temperature. For the most part, a bar slightly over-size was used to permit a skin cut to provide a test section 0.505 in. in diameter.

TABLE I.—EFFECT OF VARIOUS HEAT TREATMENTS ON THE PROPERTIES OF SAND-CAST EM42 ALLOY

Heat Number	Analyses, per cent		Heat Treatment[b]	Test Temperature, deg. Fahr.	Tensile Properties[a]				Creep Properties at 600 F.—2000 psi. Load					
	Cerium	Manganese			Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Elongation, in 2 in., per cent	Reduction of Area, per cent	Duration, hr.	Initial Deformation, per cent	Minimum Creep Rate, per cent per hr.	Deformation at End of 120 hr., per cent	Total Deformation, per cent	Contraction on Release of Load, per cent
A4932	4.25	1.51	Aged	70		13 850	...	0.8	...	...	...	...	...	...
A4931	4.45	1.50	Aged	600	8175	12 900	19.2	25.21	304.0	0.041	0.00035	0.099	0.158	0.037
A4932	4.25	1.51	A and S	70		13 850	0.8[c]	0.4[c]	...	...	...	...	...	...
A4930	4.44	1.50	A and S	600	7050	10 300	33.0	35.1	286.0	0.047	0.00027	0.105	0.185	0.053
A4929	4.40	1.49	HTA	70		16 300	1.0	0.8[c]	...	...	...	...	...	...
A4931	4.45	1.50	HTAS	600	6700	9 575	38.5	52.8	311.0	0.040	0.00008	0.075	0.082	0.042
A4929	4.40	1.49	HTQA[d]	70		16 350	1.0	1.3	...	...	...	...	...	...
A4928	4.43	1.48	HTQAS[d]	600	6600	9 700	31.7	45.5	311.0	0.045	0.00022	0.090	0.140	0.035
A5111	4.03	1.61	HTA[d]	70		16 450	1.0	0.4	...	...	...	...	...	...
			HTQAS[d]	600	6625	9 925	30.0	38.2	...	...	...	...	...	...
A4929	4.40	1.49	HTQA[e]	70		17 250	1.0	1.4	...	...	...	...	...	...
A4928	4.43	1.48	HTQAS[e]	600	6100	9 275	22.5	37.6	314.0	0.044	0.00015	0.085	0.181	0.039

[a] Average of two tests.
[b] Aged—Heated for 16 hr. at 400 F. and air cooled.
A and S—Aged and stabilized at 650 F. for 24 hr. after the above aging treatment.
HTA—Solution heat treated at 1040 F. for 24 hr., air cooled, and aged 16 hr. at 400 F.
HTQA—Solution heat treated at 1040 F. for 24 hr., quenched in either water or oil, and aged at 400 F. for 16 hr.
HTQAS—Same as above, but stabilized 24 hr. at 650 F. following the aging treatment.
HTAS—Same as HTA, but stabilized 24 hr. at 650 F. following the aging treatment.
[c] One test value.
[d] Water quenched.
[e] Oil quenched.

Heat Treatment:

As indicated previously, the data from the Dow Chemical Co. showed definitely that a solution treatment followed by an aging treatment produced the structural condition having the greatest resistance to creep at elevated temperatures. As a result, the effects of heat treatment were not investigated in detail. Table I shows the effects of heat treatment on the tensile properties at room temperature and 600 F. and upon the resistance to creep of EM42 alloy at 600 F.

Examination of the data in this table shows the following:

1. A solution and aging treatment increases the room-temperature tensile properties.
2. At 600 F., the highest tensile strength and yield strength are obtained in the cast and aged conditions. Aging and then stabilizing the unheat-treated castings reduce the tensile strength and yield strength at 600 F. to some extent. Heat treating, aging, and stabilizing still further reduce the tensile strength and the yield strength at 600 F.
3. Quenching from the heat-treating temperature into cold water or oil instead of air cooling produces a negligible effect on the tensile properties at room temperature and at 600 F.
4. At least for these particular test bars

loaded at 2000 psi., the heat treatment has had a relatively little effect upon the minimum creep rate at 600 F., but those bars which have been heat treated, aged, and stabilized appear to have slightly lower minimum creep rate than the bars which were aged only, or aged and stabilized. A greater beneficial effect of heat treatment probably would be apparent at lower testing temperatures.

The anticipated service life of the cast part was assumed to be of substantial length, and the anticipated operating service temperature was assumed to be 600 F. Obviously, under these conditions, the experimental alloys to be evaluated should be stabilized so that structural changes during the course of the test would not occur. Because exposure to the service temperature generally has an adverse effect on the tensile properties, it was reasoned that the alloy should be tested after it had been subjected to such an exposure. Since stabilization at the temperature of test requires a very long time, the test bars were stabilized at slightly higher temperature for shorter periods according to the following schedule:

1. Those tested at 300 F. were placed in the HTA condition, namely, in the heat-treated and aged state.
2. Those tested at 400 F. were first heat treated and aged, then annealed for 24 hr. at 500 F. prior to testing.
3. Bars tested at 500 F. were first heat treated and aged, and then annealed at 575 F. for 24 hr. prior to testing.
4. Bars tested at 600 F. were heat treated and aged, then stabilized for 24 hr. at 650 F. prior to testing.

The experimental compositions were tested at 600 F. only, and were, therefore, stabilized as indicated under item 4 above. AZ63, EM61, and the experimental alloy of optimum composition were also tested at 300, 400, and 500 F. as well as at 600 F. and were given the appropriate stabilizing treatments, before testing.

AZ63 alloy was heat treated and aged in accordance with commercial practices. The magnesium-cerium alloys were heat treated for 20 hr. at 1000 to 1040 F. and aged 16 hr. at 400 F. Those tested at elevated temperatures were then stabilized for the time and temperature indicated above. In some instances, because of the lowered solidus line, it was necessary to heat treat the experimental composition at a temperature slightly lower than 1040 F.

High-Temperature Tension Tests:

The screening tension test for the experimental alloys was conducted at 600 F. However, AZ63, EM61, and the experimental alloy of optimum composition were tested at room temperature, 300, 400, 500, and 600 F. The high-temperature test consisted of placing a specimen in a specially calibrated furnace mounted on the testing machine. The furnace was calibrated and adjusted so that the temperature variations between seven thermocouples were only ±5 F. After the specimen reached temperature, the load was applied to produce a cross-head movement of 0.01 in. per min. per in. of gage length until the 0.2 per cent yield strength was reached. The rate of loading was then increased to 0.03 in. per min. per in. of gage length until rupture occurred. All the bars were stabilized prior to the testing operation. The stabilized bars were inserted in the furnace, maintained at the testing temperature, and the furnace temperature again brought to equilibrium. This required 20 to 30 min., after which the specimen was maintained at temperature 30 min. and the test bar broken. Stress-strain curves were obtained on each specimen, thus providing proportional limit, modulus of elasticity, and yield strength data.

Creep and Stress-Rupture Tests:

The experimental alloys were subjected to a screening creep test at 600 F.

using a 2500 psi. load. However, considerably more complete creep data were obtained on AZ63, EM61, and the experimental alloy of optimum composition.

Both stress-rupture and creep tests were made with the same equipment and with only minor changes in testing technique. The equipment used is illustrated by Fig. 1, which shows a group of four creep or stress-rupture units. Each testing unit is equipped with a chromium-plated copper- or steel-shell furnace 6 in. in diameter and 18 in. long, wound with No. 14-gage Chromel A wire and insulated with Sil-O-Cel. These furnaces may be used for creep or stress-rupture tests up to 1800 F., but 600 F. has been the maximum temperature employed in this investigation. Small windows are provided at both the front and back of the furnace for measuring the deformation of the test specimen by optical means.

A standard 0.505-in. test specimen and calibration specimens are shown by Fig. 2. The temperature gradient in the furnace can be controlled and changed by means of external shunts along the furnace winding. For this purpose, the calibration specimen shown in Fig. 2 is used and the furnace shunted so that the maximum temperature variations measured by thermocouples at positions T_i, C_i, B_i, T_o, C_o, B_o do not exceed ±3 F. at the test temperature. In some instances, even smaller variations are obtained. All the temperature variations

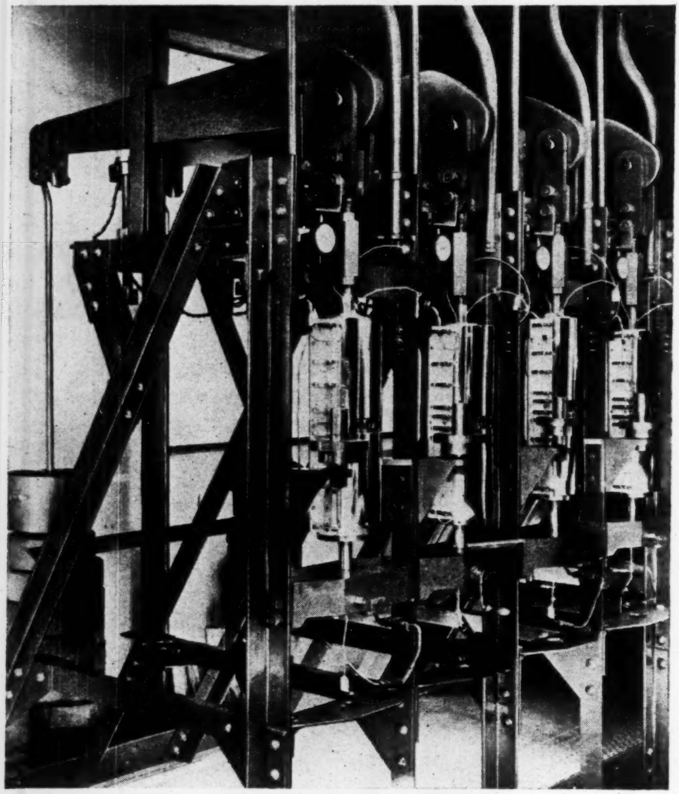
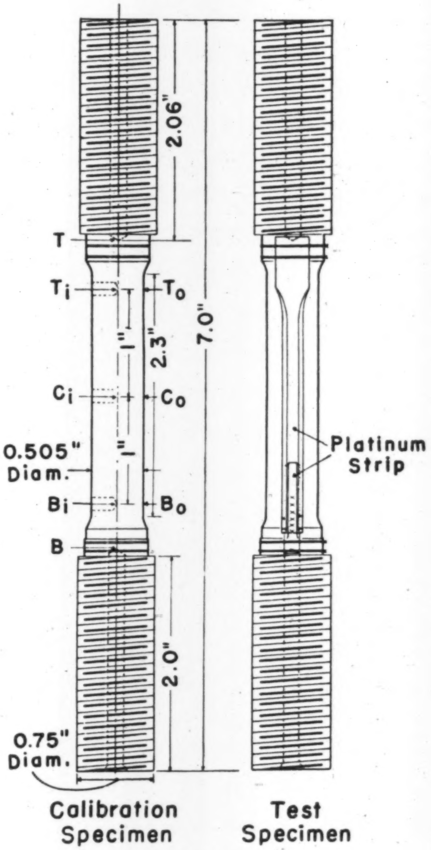


FIG. 1.—Creep or Stress-Rupture Units.

are kept below the maximum allowable by the A.S.T.M. Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41).[4] During the actual creep or stress-rupture tests, thermocouples are located at positions at T_o and B_o and are used for adjusting the test temperature. Thermocouples are also placed at positions T and B, the control couple at B, and a recorder thermocouple at T. These thermocouples can be replaced while the test is in progress.



Calibration Specimen Test Specimen

FIG. 2.—Calibration and Test Specimen.

The load is applied to the test specimen by means of a lever arm with a 9 to 1 ratio.

When stress-rupture tests are being made, a switch is mounted on the lever-arm stop under the lever arm to cut off the power to the furnace and stop the clock when the test specimens break. The clock measures the duration of the test to 0.1 hr.

TABLE II.—TYPES OF ALLOYS INVESTIGATED.

Base[a]	Addition	Number of Heats
AZ63—6 per cent Al—3 per cent Zn	None	10
4 to 15 per cent Ce	None	9
3 to 6 per cent Ce	Zr	9
3 per cent Ce	Zr + Zn	
4 to 10 per cent Ce	Zr + Cd	21
4 to 10 per cent Ce	Mn	35
6 per cent Ce	Mn with Ag, Al, B, Ba, Be, Bi, Ca, Cd, Cr, Li, Ge, Cu, In, Pb, Sb, Si, Sn, Te, Ti, Tl, Zn, Mo, As, Cb, Hg, K, Na, P, Sr, Ta, W	122
11 per cent Ce	Mn + Be	1
6 to 10 per cent Ce	Mn + Zr, Mn + Co	33
6 to 10 per cent Ce	Mn with Ni + Co, Ni + Zr	6
5 to 10 per cent Ce	Mn with Zr + Ca, Zr + Cd, Zr + Ge, Zr + Ge	13
6 per cent Ce	Mn with Be + Al, Be + Zr, Be + Cd, Be + Cu	4
6 per cent Ce	Mn with Ni + Ti, Ni + Zr, Ni + Cu + Zr, Ag + Co + Zr, Ni + Mo, Ni + Ba, Ni + Be, Ni + Ca, Ni + Ge, Ni + P, Ni + Sr, Ni + Tl, Ni + W, Ni + V, Ni + Bi	38
10 to 11 per cent Ce	Mn with Co + Zr, Co + Be + Zr	6
3 to 6 per cent Ce	Ni, Mn + Ni, Co + Ni	37
6 per cent Ce, 0.6 per cent Mn, 0.2 per cent Ni	Tl + P, Tl + Ge, W + V, Be + W, W + Zr, Be + Sr + Tl, Ba + Sr + Tl, Sr + W + Be, Be + W + Zr	13
6 to 10 per cent Ce	Ag	2
Mg	1.4 per cent Mn, 1.0 per cent Ni, 1.0 per cent Cu, 3.5 per cent Co	1
Mg	1.80 per cent Mn, 1.07 per cent Ni, 0.60 per cent Zr, 0.35 per cent Co	1

[a] Per cent Ce indicates per cent rare earths. All alloys were magnesium base.

For the measurement of deformation, platinum strips are used which are attached to the shoulders of the specimen, one at each end of the gage length. Figure 2 shows the strip in position on the test specimen. A series of very fine cross marks is ruled on each strip. Changes in length of the test specimen are measured

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[4] 1949 Book of A.S.T.M. Standards, Part 1, p. 1281.

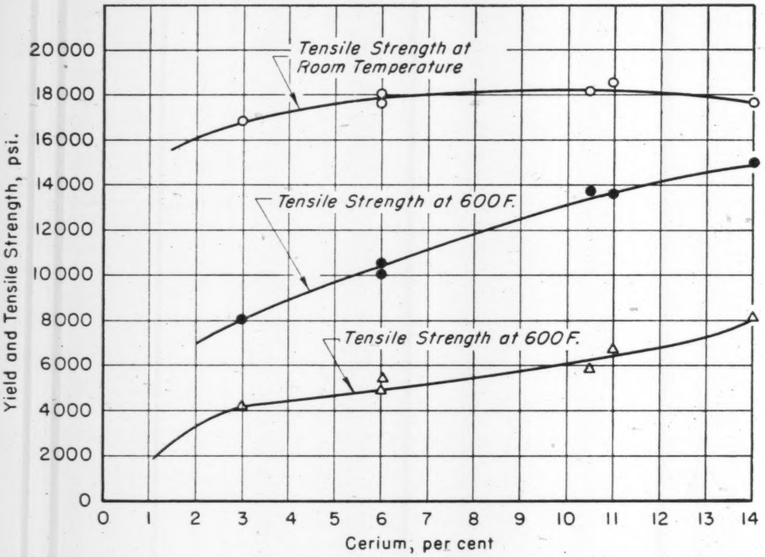

FIG. 3.—Effect of Cerium on the Properties of Mg-Ce Cast Binary Alloys in the HTA Condition at Room Temperature and in the HTAS Condition at 600 F.

by determining the change in distance between the two chosen cross marks, one on each strip. The microscope with which the deformation readings are made has an eyepiece fitted with a filar micrometer and is mounted on a graduated screw. Calibrations show the smallest division of the filar eyepiece to read 0.00005 in., which, on the gage length of about 2.3 in., provides readings slightly over 0.00002 in. per in., or within about 0.002 per cent.

Deformation readings are usually made daily by two observers, and the time-deformation data illustrated in this report are the averages obtained by the two observers.

ALLOY DEVELOPMENT

When considering the effects of alloy addition, it is important to remember that the screening tension and creep tests were conducted at 600 F. The results cannot be safely compared with those obtained at a lower temperature of test. Thus, a composition which is optimum at 600 F. may not be the best at 400 F.

A considerable number of alloys containing cerium (rare earths) have been studied. The effects of cerium in the range of 3 to 14 per cent are illustrated by Fig. 3. These effects of cerium have been shown in greater detail by others (5, 6, 12). It is quite evident that the tensile and yield strengths at 600 F. increase with cerium content. Cerium also has a considerable effect upon the creep properties, particularly on total deformation, as shown by the following data:

Cerium, per cent	Condition	Creep Rate, per cent per hr.		Total Deformation in 120 hr., per cent	Duration of Test, hr.
		Minimum	Final		
3........	HTAS	0.004	0.025	5.24	117
6........	HTAS	0.004	0.004	1.40	140
10.5......	HTAS	0.0058	0.0058	1.24	173

Six per cent cerium was selected as the optimum content, namely, as a compromise between better high-temperature properties on the one hand and lower costs and lower density on the other. As a result, the major portion of the alloy

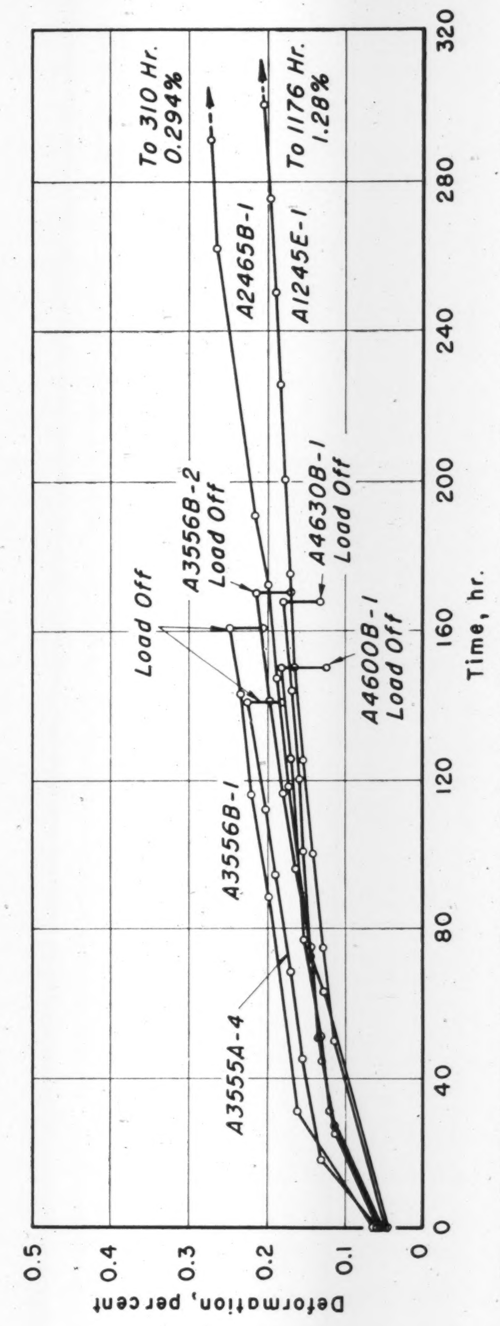

FIG. 4.—Deformation-Time Curves Obtained on Six Heats of EM62 Alloy at 600 F. and 2500 psi. Minimum Creep Rates Varied from 0.00075 per cent per hr.

development was carried out on the magnesium-6 per cent cerium base.

Manganese also has a fairly marked effect on the properties of the magnesium-cerium alloys. For example, E6, containing 6 per cent cerium and EM62 alloy, containing 6 per cent cerium and 1.8 manganese, have the following properties:

	Condition	E6	EM62
Tensile strength at room temperature, psi.	HTA	18 000	16 000
Tensile strength at 600 F., psi.	HTAS	10 000	10 500
Yield strength at 600 F., (0.2 per cent offset) psi.	HTAS	5 000	6 500
Minimum creep rate, 2500 psi. load, 600 F., per cent per hr.	HTAS	0.004	0.0007

The effects of manganese on the creep and tensile properties of a 6 per cent cerium-base alloy are also shown by the following data:

Manganese, per cent	Creep Properties, 2500 psi., 600 F.				Tensile Properties			Grain Size, in.
	Creep Rate		Total Deformation in 120 hr., per cent	Duration of Test, hr.	Room Temperature	600 F.		
	Minimum, per cent per hr.	Final, per cent per hr.			Tensile Strength, psi.	Yield Strength, 0.1 per cent, psi.	Tensile Strength, psi.	
0	0.004	0.004	1.4	139.6	17 650	4 900	10 000	0.025
0.25	0.0025	0.0025	0.375	144	17 000	4 800	11 100	0.025
0.60	0.00095	0.00095	0.245	141	17 300	5 400	11 200	0.025
1.0	0.00017	0.0006	0.159	286	16 000	6 000	10 600	0.08
1.8–2.0[a]	0.0007	0.0007	0.192	140–310	16 000	6 100	11 000	columnar

[a] 5 tests on different heats.

It is quite evident from these data that manganese has a somewhat beneficial effect upon creep rate and tensile properties at 600 F. Another important observation is the increasing grain size produced by increasing the manganese content above 1 per cent. Relatively fine grains are obtained at lower manganese content. These castings were poured at 1400 to 1420 F. As a result of these observations, a considerable amount of work was done on alloys containing 6 per cent cerium (rare earth) and 0.6 to 1.6 per cent manganese.

The experimental alloys which were made up and subjected to the routine screening tests totaled about 350. All of these alloys were subjected to tension tests at room temperature and at 600 F. and to metallographic examination to determine their grain size, soundness, and response to heat treatment. In addition, a very large number of those which appeared to have outstanding tensile properties at 600 F. were also subjected to the standard creep test consisting of a 2500 psi. load at 600 F. This test was continued for approximately 1 week, at which time the test was abandoned unless the creep rates were low, indicating a beneficial effect of the alloy addition. In such instances, the creep test was continued for about 2 weeks.

Because creep resistance at 600 F. was an essential consideration, some importance was attached to the standard creep test. The reliability of the test became significant. The remarkable reproducibility of the time-deformation data obtained in the standard creep test is illustrated by Fig. 4. This figure shows the time-deformation values for several heats of the EM62 composition prepared at various intervals during the course of the three years of experimental work. These results attest to the reliability of the standard creep test as a means of screening the experimental cast alloys.

The types of alloys investigated are indicated by the data in Table II. For the most part, the added elements were low in amount, usually over a range up to 1 per cent. Readily soluble elements such as silver, aluminum, cadmium, copper, and zinc were added in greater amounts. Difficultly soluble elements such as beryllium, tantalum, titanium, molybdenum, tungsten, and vanadium were added in very small amounts or the amount dissolved by the melt was unknown, as indicated earlier.

Figure 5 is representative of a very large number of time-deformation curves for the various experimental alloys. Some of the additions to the EM62 composition have a beneficial effect, others a harmful effect on the resistance to creep at 600 F. Alloy additions which produced a time-deformation curve appreciably under that representative of the EM61 composition were, of course, of greatest interest.

The general procedure consisted of evaluating the effects of individual elements and then attempting to combine beneficial elements into more complex alloys. The individual effects of the additions to either a 6 per cent cerium base or 6 per cent cerium-0.0 to 1.8 per cent manganese base are described below.

Zirconium:

Zirconium was one of the first elements investigated in the early part of the work in 1945. The outstanding effect of this element is grain refinement. Presumably, because of this grain refinement, it would reduce difficulties with hot cracking in the foundry. EM61 composition poured from about 1400 F. will normally have a grain size of about 0.20 in. When a few hundredths of one per cent of zirconium is added to this composition, the grain size becomes, on the average, about 0.02 in. As already noted, manganese causes a considerable coarsening of the grain of the alloy poured at 1400 F. If the manganese is omitted, Composition E6 results and the grain size of test bars of this alloy poured from about 1400 F. is about 0.02 in. The addition of a few tenths per cent of zirconium will reduce the grain size of the E6 base alloy poured from 1400 F. to approximately 0.002 in. It should also be noted that, in the absence of manganese, higher zirconium contents can be obtained in the alloy because of the higher solubility of the zirconium in the melt. Although zirconium did not appear to have beneficial effects on the tensile properties or creep resistance of the E6 or EM61 base compositions at 600 F., considerable effort was made to employ it because of the desirable effects of grain refinement on casting properties. It is interesting to note that grain refinement produced by lowering the pouring temperature from 1400 to 1280 F. had relatively little effect upon the resistance to creep or the tensile properties at 600 F., but such grain refinement did produce a slight increase in room-temperature tensile properties, particularly in tensile strength and elongation. Grain refinement produced by zirconium additions generally lowered the creep resistance to some extent or made it more erratic. In general, it was found that the effects of zirconium additions to manganese-cerium base alloy were variable, particularly when manganese was present.

Calcium:

Calcium lowered the solidus temperature and, therefore, made lower heat-treating temperatures mandatory. When approximately 0.3 per cent was added, a marked reduction in oxidation of the melt occurred in the pot. However, limited creep data indicated that such additions were harmful to the creep resistance of the EM61 base.

Other Elements:

Most of the elements added had little effect upon the tensile properties of the EM62 base at room temperature or at 600 F. However, copper appeared to be harmful, and aluminum, and possibly zinc, were slightly harmful to the tensile

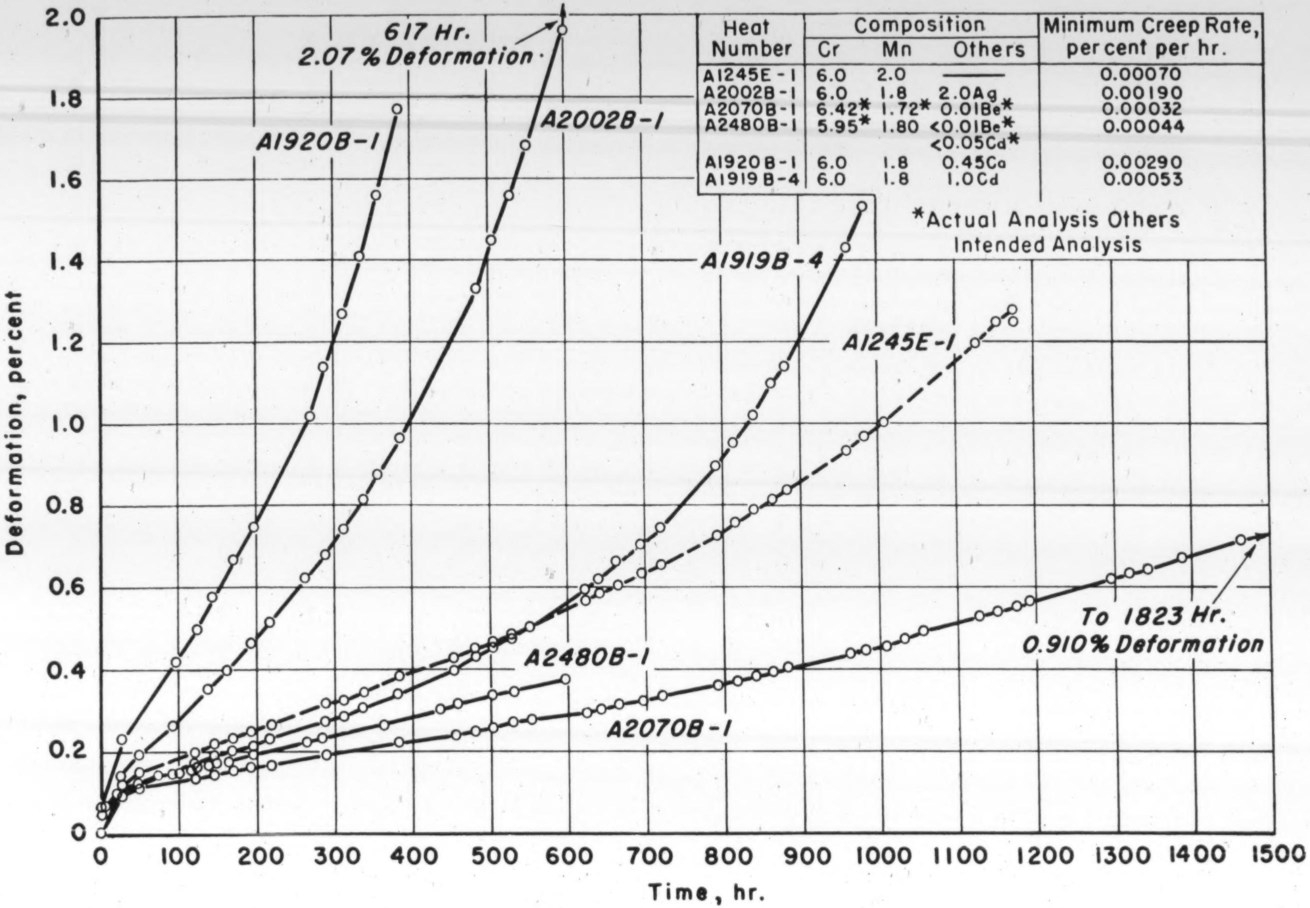

FIG. 5.—Time-Deformation Curves of EM62 and Experimental Alloys at 600 F. and 2500 psi. Load.

properties at 600 F. Small amounts of nickel have beneficial effects, particularly on the tensile properties of the 6 per cent cerium-0.8 per cent manganese alloy at 600 F. The following additions appear to have some beneficial effect on the creep resistance of the EM62 base:

Antimony
Barium
Beryllium
Bismuth
Cadmium
Chromium
Columbium
Lead
Molybdenum
Nickel
Strontium
Tantalum
Tellurium
Thallium
Tin
Silicon

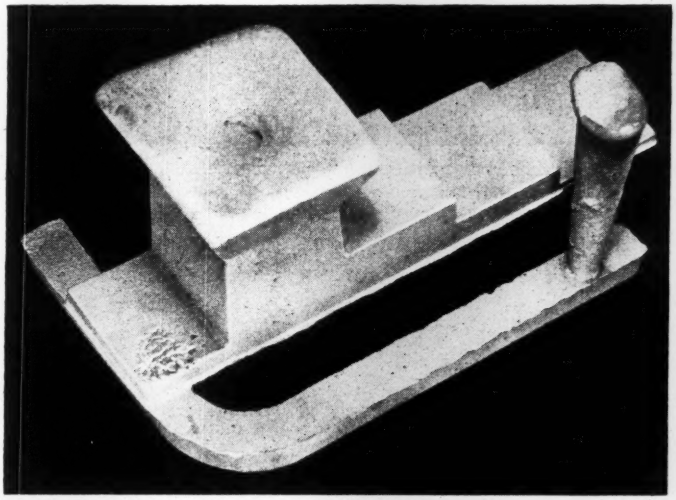

FIG. 6.—Photograph Showing Method of Gating and Risering of Step-Bar Casting.

Of these elements, tantalum, silicon, chromium, columbium, and molybdenum are fairly insoluble and difficult to add to the melt and obtain consistent results. Of the remaining elements, cadmium, beryllium, nickel, strontium, thallium, and barium are probably the most promising. Consequently, considerable effort was made to employ various combinations of these elements in the magnesium-cerium-manganese base.

Preferred Composition:

Because a coarse-grained alloy may be quite difficult to cast in the foundry as a result of hot cracking, considerable effort was devoted to the attainment of an alloy which tended to be fine grained but at the same time possessed good tensile properties and creep resistance at 600 F. As noted before, a reduction in manganese effects a considerable grain refinement. When, however, manganese is reduced, the tensile properties at 600 F. are also reduced somewhat. It was found, however, that, if the manganese was reduced to about 0.8 per cent and 0.2 per cent nickel added, a relatively fine-grained casting was produced while the tensile properties at 600 F. were even higher than those of the EM62 composition. Further grain refinement was obtained by small additions of zirconium or tungsten. As a result, the following composition was selected as optimum and recommended for field tests:

Cerium, per cent	6
Manganese, per cent	0.8
Nickel, per cent	0.2
Tungsten, per cent	0.02

The composition limits have not been established. Furthermore, the possibility of reducing the cerium content has not been fully explored nor have the full potentialities of using small amounts of beryllium and zirconium been fully evaluated. Beryllium appears to improve the creep resistance of the alloy, though it tends to coarsen the grain while zirconium tends to produce fine-grained castings. However, since the usefulness of these two additions to an alloy containing 6 per cent cerium, 0.8 per cent manganese, and 0.20 per cent nickel has not been fully evaluated, they were omitted from the recommended composition. Considerable study was made of the alloy of the above optimum composition and further data will be presented on it.

FIG. 7.—Intended Composition: 6.00 per cent Ce, 2.00 per cent Mn.
Pouring Temperature 1500 F. (Heat No. A-4494).

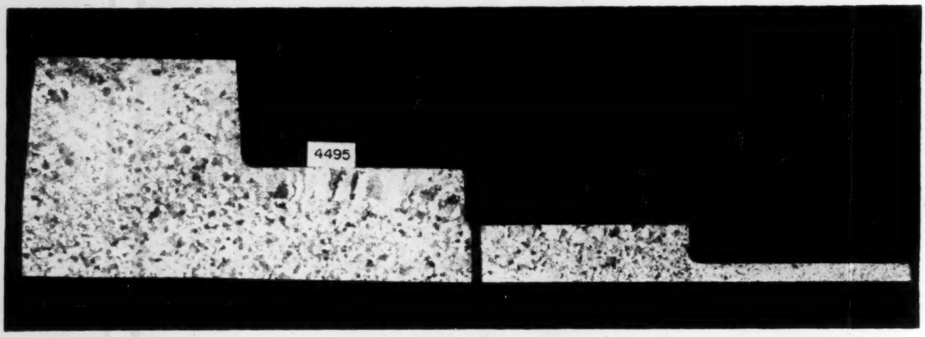

FIG. 8.—Intended Composition: 6.00 per cent Ce.
Pouring Temperature 1500 F. (Heat No. A-4495).

Grain-Size Studies:

Grain-size studies were made using the casting shown in Fig. 6. This casting is 3 in. wide and has sections 3, $1\frac{1}{2}$, $\frac{3}{4}$, and $\frac{1}{4}$ in. thick. Each section is 3 in. long. This casting was sectioned longitudinally through the center, and the sections polished and etched to reveal their grain sizes. Figure 7 shows the EM62 compositions as poured from 1500 F. While the photograph is reduced to nearly half size in reproduction, the very coarse grain is obvious. Figure 8 shows the E6 composition also as poured from 1500 F. The substantial reduction in grain size produced by eliminating the manganese is quite evident. Note, however, the fairly coarse columnar grains on the cope side of the $1\frac{1}{2}$ in. and $\frac{3}{4}$-in. sections.

Figure 9 shows the grain size of the casting containing 6 per cent cerium, 0.6 per cent manganese, and 0.2 per cent nickel poured from 1500 F. As compared with the E6 alloy, it is slightly finer grained.

A casting containing 6 per cent cerium, 0.6 per cent manganese, 0.2 per cent nickel, and 0.5 per cent zirconium poured from 1420 F. is shown by Fig. 10. Comparing this photograph with Fig. 9, representing a casting which does not contain zirconium, it is quite evident that the zirconium addition has produced some grain refinement. The experimental creep results obtained on an alloy of this composition indicated that it was somewhat variable in resistance to creep. However, further study of this composition could quite probably eliminate this difficulty.

A casting containing the tentative optimum composition of 6 per cent cerium, 0.6 per cent manganese, 0.2 per cent nickel, and 0.02 per cent tungsten, poured from 1415 F., is illustrated by Fig. 11. It is evident that this is a fairly fine-grained casting though not quite so fine as the zirconium-bearing alloy represented by Fig. 10.

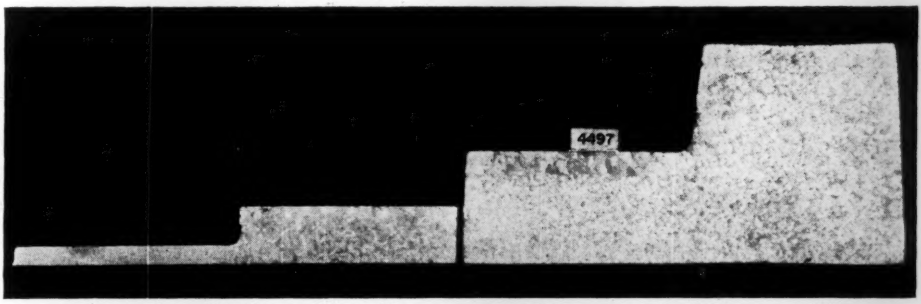

FIG. 9—Intended Composition: 6.00 per cent Ce, 0.60 per cent Mn, 0.20 per cent Ni.

Pouring Temperature 1500 F. (Heat No. A-4497)

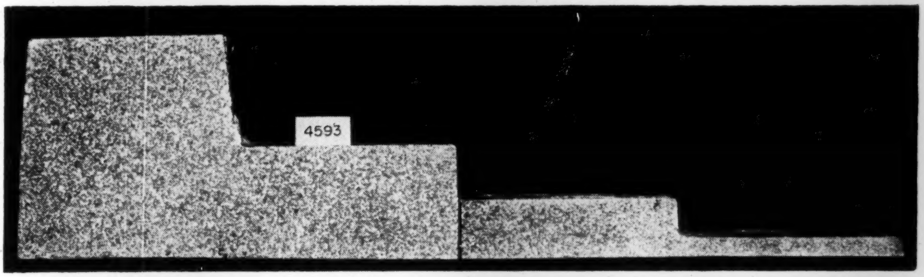

FIG. 10.—Intended Composition: 6.00 per cent Ce, 0.90 per cent Mn, 0.20 per cent Ni, 0.70 per cent Zr.

Zirconium added to magnesium containing 0.90 per cent manganese. Pouring temperature 1420 F. (Heat No. A-4593).

FOUNDRY CHARACTERISTICS OF MAGNESIUM-CERIUM ALLOYS

The foundry characteristics of magnesium-cerium alloys are of considerable interest. Experience has shown that the magnesium-cerium alloys are quite similar in this respect to the standard zinc-aluminum alloys, AZ63 and AZ92. However, a few peculiarities were noted.

When misch metal is added to the magnesium melt, a few heavy nonmetallic particles appear to form and tend to settle to the bottom of the crucible. However, if they are not given an opportunity to settle out of the melt, as when the melt is stirred and then poured quickly into molds, these heavy nonmetallic particles will enter the mold cavity and can be found in the castings, particularly on the drag surface. No effort was made to identify these heavy nonmetallic particles. It is apparent, however, that they need cause no serious difficulty in the foundry if they are permitted to settle to the bottom of the pot before the melt is transferred to a pouring ladle.

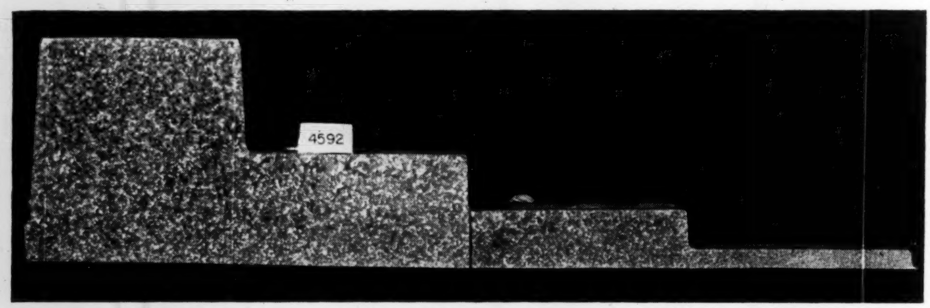

FIG. 11.—Intended Composition: 6.00 per cent Ce, 0.60 per cent Mn, 0.20 per cent Ni, 0.02 per cent W.

Pouring temperature 1415 F. (Heat No. A-4592).

Another characteristic of the magnesium-cerium alloys is their greater tendency to form dross as the result of turbulence during the pour. Therefore, very good gating practices must be employed to avoid such turbulence. This, of course, is a very important subject, too large to be discussed in this paper. It should be emphasized, however, that gating practices adequate for the ordinary magnesium alloys may be disastrous when casting magnesium-cerium alloys.

Another peculiarity of the magnesium-cerium alloys which also characterizes the standard alloys, but to a lesser extent, is the tendency of a dark discolored area to occur sometimes on the cope surface of the casting. These dark areas appear to be a result of local sand reaction where the metal appears to penetrate the sand. The defects were successfully eliminated by avoiding soft spots in the surface of the green sand and cores and avoiding too great an impact of the metal with the mold surface during the pour.

Another characteristic of the magnesium-cerium alloys is their hot shortness. This characteristic apparently is a serious limitation in the case of the EM61 or EM62, compositions which tend to be very coarse-grained at the high pouring temperatures. Several castings were made with the experimental alloy of optimum composition. However, these castings were simple ones: test bars, plates, small frames, and covers. All of these castings could be made successfully and no difficulty with hot cracking was encountered. Commercial foundry tests on complex aircraft motor castings encountered no difficulty with hot cracking. It is recommended, however, that good gating and melting practices be employed and that pouring temperatures not exceed 1425 F.

The cerium alloys, particularly those containing 6 per cent, are not susceptible to the formation of microporosity, as is AZ63 alloy. This is entirely within expectations, however, because the solidification range is narrow, and there is a fairly large quantity of liquid which freezes at constant temperature at the end of solidification.

Exact measurements of the fluidity or mold-filling capacity of the alloys are fairly difficult to make. Consequently, detailed studies of this kind were not made. Limited experience indicates, however, that, because of the higher melting point range of the magnesium-cerium alloys, they require, for a given set of foundry conditions, higher pouring temperatures than does AZ63 alloy.

HIGH-TEMPERATURE OXIDATION OF MAGNESIUM-CERIUM ALLOYS

The resistance of the experimental alloy of optimum composition to oxidation at 600 and 700 F. was not studied. However, these studies were made on the EM62 composition. It was found that some oxidation of the cerium-rich phase occurs at 600 and 700 F. during prolonged exposures to these temperatures. Figures 12 and 13 illustrate the appearance of the surface after such oxidation. These specimens were exposed in air of the high humidity typical of the summer months in Central Ohio.

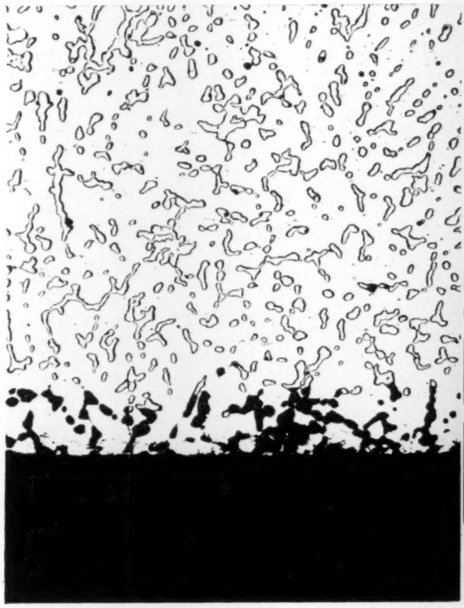

FIG. 12.—Photomicrograph of a Section Taken from a Creep Specimen Tested at 600 F. for 642 hr. (× 100).

Surface oxidation has started along the grain boundaries. EM 62 alloy.

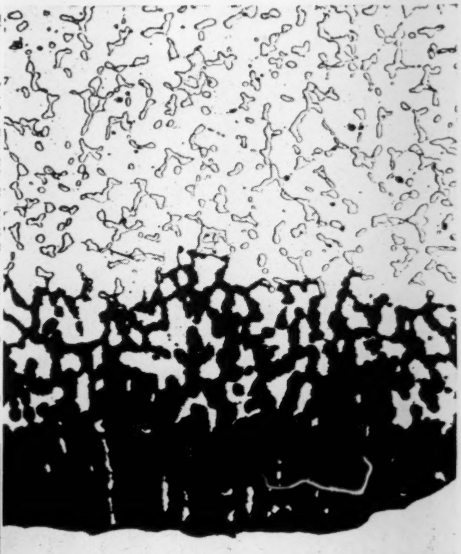

FIG. 13.—Photomicrograph of a Section Taken from a Creep Specimen Tested at 700 F. for 477 hr. (× 100).

Oxidation has penetrated deeply into the specimen. EM 62 alloy.

This high-temperature oxidation appeared to be serious if long service exposures to 600 F. were contemplated. Some effort, therefore, was devoted to the development of protective coatings. Several methods were tried, but the only one found successful was as follows:

First Step.—The specimen was degreased in a bath containing the following composition:

Trisodium phosphate	240	g.
Sodium carbonate	240	g.
Distilled water	8.0	l.

The bath temperature was maintained at

180 to 240 F. and the immersion was for 5 to 15 min., using a current density of 15 amp. per sq. ft. After degreasing, the specimen was rinsed in cold water.

Second Step.—The specimen was then treated in a fluosilicic acid bath of the following composition:

Hydrofluosilicic acid (H_2SiF_6)	250 ml.
Distilled water	750 ml.

Titanium potassium oxalate to produce saturation.

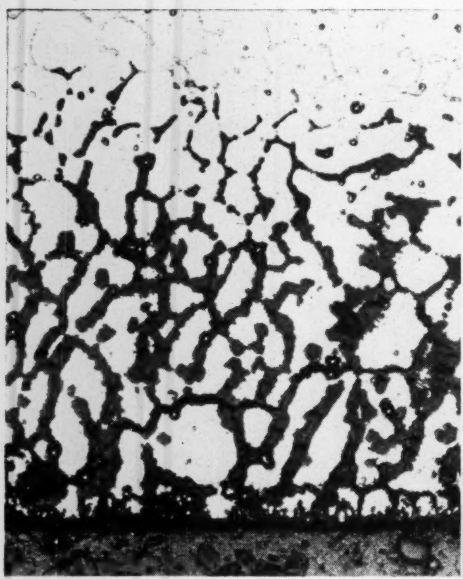

FIG. 14.—Photomicrograph of a Section Taken from an Uncoated EM62 Alloy Specimen After Approximately 900 hr. Exposed at 700 F. (× 100).

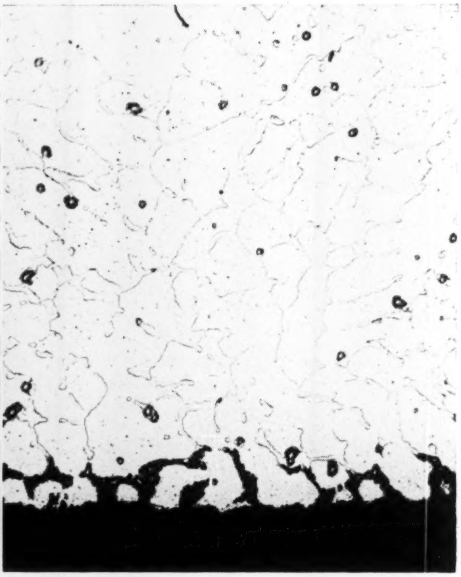

FIG. 15.—Photomicrograph of a Section Taken from a Cast EM62 Alloy Test Bar Coated by the Fluosilicic Acid Treatment Exposed approximately 900 hr. at 700 F. (× 100).

This bath was used at room temperature and the specimens were immersed for 10 min. After this treatment, they were subjected to a cold-water rinse, then a hot-water rinse, and then dried in an air blast.

Figures 14 and 15 show photomicrographs of EM62 alloy exposed for 900 hr. at 700 F. The specimen represented by Fig. 14 was uncoated, while the one represented by Fig. 15 was subjected to the fluosilicic acid treatment as described above. The marked improvement is quite evident, and this method of protection of magnesium-cerium alloys exposed for prolonged periods at elevated temperatures merits further investigation.

THE PROPERTIES OF ALLOYS

As indicated earlier, the following composition was selected for further study:

Cerium, per cent	6
Manganese, per cent	0.8
Nickel, per cent	0.2
Tungsten, per cent	0.02

This alloy is very much finer grained than the EM61 or EM62 compositions, even when it is poured at high temperatures into heavy sections. In addition, it has tensile properties at 600 F. which are higher and creep resistance at least equal to that of the EM62 composition. The alloy of optimum composition is finer grained than E6 and has substantially

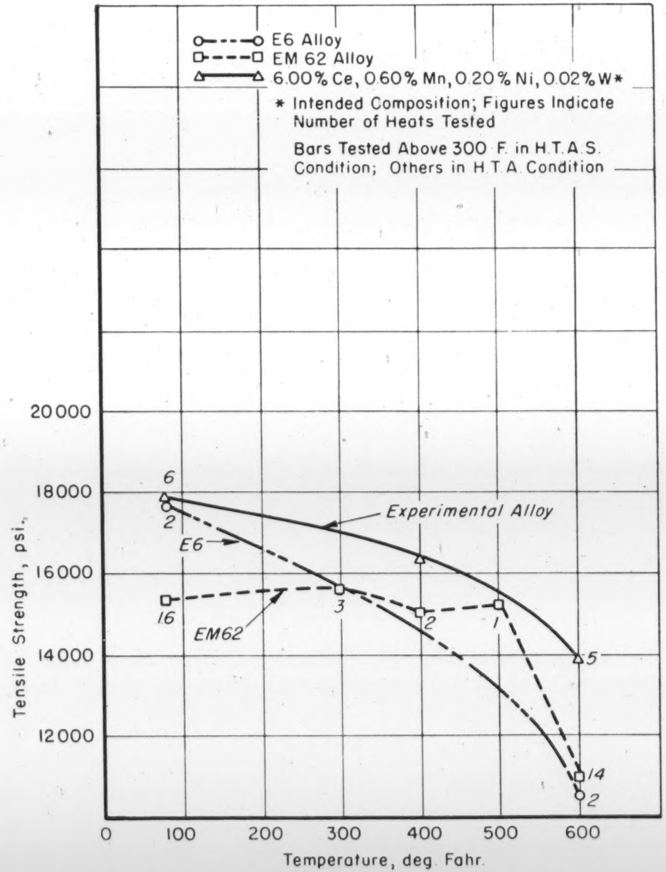

FIG. 16.—Comparison of Tensile Strength of E6, EM62, and an Experimental Alloy at Temperatures of 70 to 600 F.

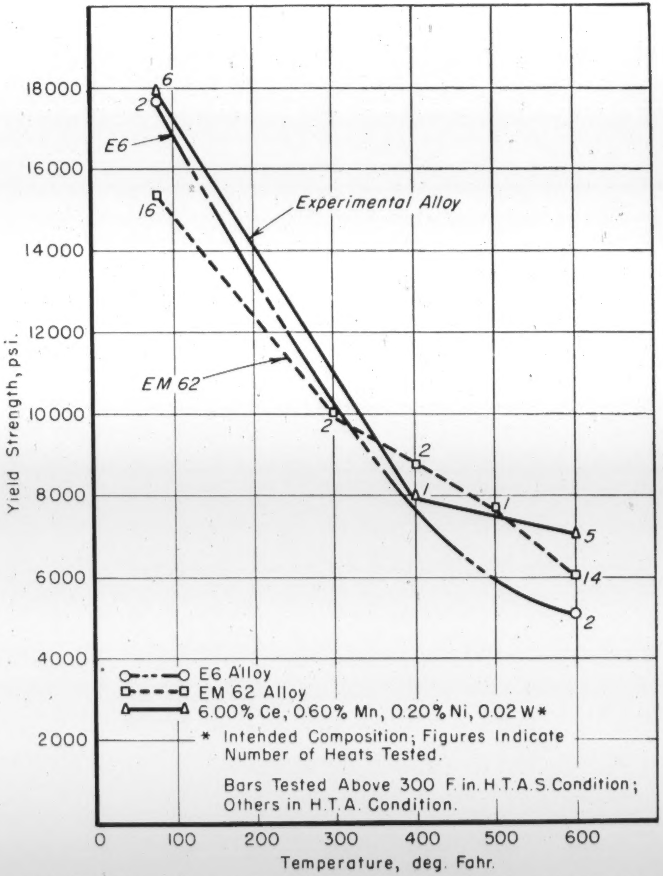

FIG. 17.—Comparison of Yield Strengths of E6, EM62, and Experimental Alloy over a Range of Temperatures.

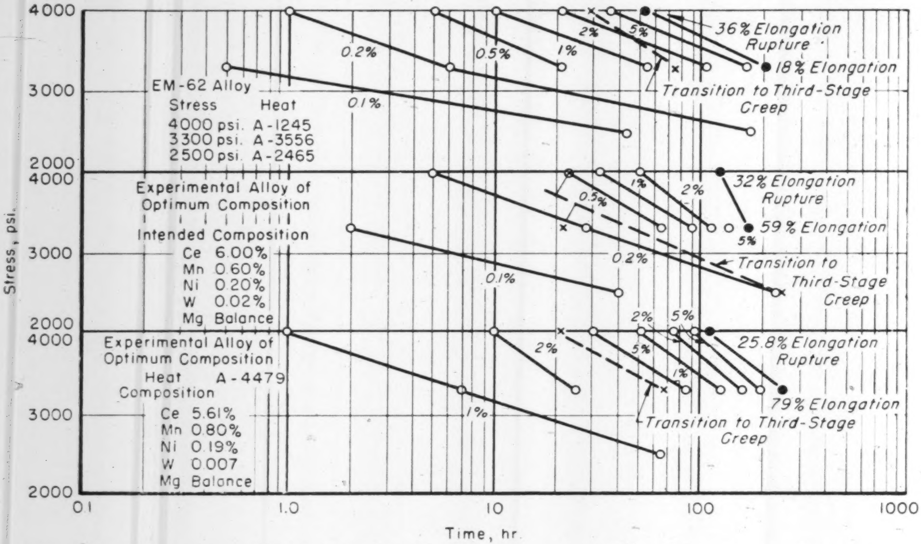

FIG. 18.—Comparison of Design Curves of EM62 Alloy and Two Heats of the Experimental Alloy of Optimum Composition at 600 F.

TABLE III.—COMPARISON OF THE AVERAGE PROPERTIES OF THE EXPERIMENTAL ALLOY OF OPTIMUM COMPOSITION WITH OTHER CAST ALLOYS.[a]

Alloy	BMI alloy	EM62	E6	EK30		ZRE1 or EZ33[b]	142-T61
Condition[c]	HTAS	HTAS	HTAS	HTAS	HTA[b]	Aged	(Stabilized)
Nominal composition	Mg-6 Ce-0.8 Mn-0.2 Ni-0.02 W	Mg-6 Ce-1.8 Mn	Mg-6 Ce	Mg-3.25 Ce-0.35 Zr		Mg-2.75 Ce-2.75 Zn-0.6 Zr	Al-4 Cu-0.6 Fe-0.8 Si-1.5 Mg-2 Ni-0.2 Ti
Grain size, in. (heavy sections poured at 1450 F.)	0.04	0.4	0.12	...	...	...	0.04
Tensile strength, psi.:							
Room temperature	17 850	15 400	17 750	...	22 900	20 300	37 000
400 F.	16 375	15 000	...	...	20 000	20 100	22 000
600 F.	13 900	10 900	10 550	8 000	11 800	8 900	8 000
Tensile yield strength psi.:[d]							
Room temperature	17 850	15 400	17 750	...	16 500	12 300	32 000
400 F.	7 950	8 650	...	...	14 000	10 500	25 000
600 F.	7 050	6 000	5 100	3 950	7 700	6 200	4 000
Elongation in 2 in., per cent:							
Room temperature	1.0	0.8	0.8	...	2.4	4.0	0.0
400 F.	4.5	2.8	...	...	13.1	22.0	0.5
600 F.	36.3	30.2	43.0	75	69	64.0	10.0
C_L = load, psi., to produce 0.5 per cent deformation in 100 hr. at 600 F.	3100	3100	1700 (HTA)[b]	...	1700	1900	4900
$C_L \div$ density	1680	1680	930		940	1040	1700
Minimum creep rate, 600 F. at 2500 psi., per cent per hr.	0.0007	0.0007	0.004	Very high	...	...	0.00017
Total deformation, 120 hr. at 600 F. at 2500 psi. load, per cent	0.12 to 0.14	0.15 to 0.23	1.4	5.5	...	...	0.05

[a] Data based on a few tests only. Also see Reference (7).

[b] Tensile data on these alloys at 600 F. in the A or HTA condition are fictitiously high because they are not stabilized at test temperature.

[c] Properties of BMI alloy, EM62, and E6 at room temperature for HTA condition.

[d] 0.2 per cent offset.

better tensile properties and creep resistance at 600 F.

During the course of the experimental work, a considerable amount of tensile and some creep data were obtained on AZ63 alloy, EM62, and the alloy tentatively selected as the optimum composition. These data have been reproduced elsewhere (8). However a comparison of the tensile and creep properties of the experimental alloy and EM62 are shown by Figs. 16, 17, and 18.

SUMMARY

About 350 heats of magnesium-cerium cast alloys, most of which contained other elements, have been prepared and studied. The evaluation of the compositions was based on the following tests:

1. Tension tests at room temperature.
2. Tension tests at 600 F.
3. Creep tests at 600 F., using a 2500 psi. load.
4. Macro and microexaminations.

Six per cent cerium (rare earths) was selected for study as a compromise between cheaper and lower density alloys of lower cerium content, on the one hand, and higher strength alloys of still higher cerium content, on the other. Manganese was also employed in most of the alloys because of its beneficial effects on tensile properties and creep resistance at 600 F. The alloy of optimum composition tentatively selected is as follows:

Cerium (rare earths), per cent	6
Manganese, per cent	0.8
Nickel, per cent	0.2
Tungsten, per cent	0.02

The possibilities of lowering the cerium or employing zirconium and beryllium in this composition look promising, but these variations in compositions have not been fully evaluated.

A comparison of the properties of this alloy with four other cerium-containing alloys and the aluminum-base alloy No. 142 is shown in Table III. Examination of the data clearly shows the superiority of the experimental alloy of optimum composition. Its creep resistance at 600 F. is similar to that of EM62, but it has the advantages of having higher tensile properties at 600 F. and being very much finer grained when cast. As compared with EM62 alloy, the number of grains per unit volume is approximately one thousand times greater for the alloy of optimum composition. A marked superiority in load-carrying capacity over E6, EK30, and the English alloy, ZRE1, at 600 F. is quite evident.

Master alloys are producible which render the preparation of the above experimental alloy relatively easy. Melting in steel crucibles under a nitrogen atmosphere is recommended. Careful foundry practices are required. Prolonged exposure at 600 F. will cause the magnesium-cerium alloys to oxidize at the surface. A chemical treatment has been developed which greatly reduces this oxidation.

Acknowledgment:

The authors gratefully acknowledge the support of the Wright-Patterson Air Force Base and the Bureau of Aeronautics, who made this investigation possible and who granted permission for its publication; the valuable efforts of Ray Kaiser, who did much of the laboratory work; the guidance and encouragement of C. H. Lorig; the assistance of H. C. Cross and Ward Simmons of Battelle, who conducted the creep tests; F. W. Fink, who developed the protective coating; and C. S. Dumont, who developed the master alloy.

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(5) T. E. Leontis, and J. P. Murphy, "Properties of Cerium-Containing Alloys at Room Temperatures," *Metals Technology*, Am. Inst. Mining and Metallurgical Engrs., Vol. 13, April, 1946, p. 295.

(6) T. E. Leontis, "The Room- and Elevated-Temperature Properties of Some Sand Cast Magnesium-Base Alloys Containing Zinc," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 180, p.287 (1949).

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(9) C. M. Craighead, Webster Hodge, and L. W. Eastwood, "Aluminum 6 Per Cent Magnesium Cast Alloys for Elevated-Temperature Service," Presented to National Metal Congress, Fall, 1950.

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(11) K. Grube, J. A. Davis, and L. W. Eastwood, "Magnesium-Cerium Wrought Alloys for Elevated-Temperature Service," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 965 (1950).

(12) T. E. Leontis, *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 185, p. 968 (1949).

DISCUSSION

MR. J. W. MEIER.[1]—In view of the recent progress in the field of jet-propelled engines, the development of magnesium-base alloys suitable for elevated temperature service became very important and urgent. The authors are to be congratulated for their interesting paper and the presentation of valuable data on a very promising new casting alloy.

Experimental work on magnesium casting alloys for elevated temperature service was conducted during the past few years at the Canadian Bureau of Mines, and some of our results were presented at the Annual Meeting of the Canadian Institute of Mining and Metallurgy a few weeks ago.[2] In this connection I should like to raise a few problems concerning the use of the magnesium-rare earths alloys.

Research on magnesium-base alloys for elevated temperature service is being conducted mainly, if not exclusively, for the aircraft industry where the highest strength-to-weight ratio is the most essential characteristic for any material to be used. Therefore it seems to be advantageous to divide the range of elevated temperatures into three service condition ranges:

1. Up to approximately 250 F.—where the highest-strength Mg-Zn-Zr casting alloys, for example, alloy ZK61, should be used for all structural components.

2. From 250 F. to approximately 400 F.—where the Mg-R.E. (rare earths)-Zr or Mg-R.E.-Zn-Zr casting alloys appear to show the best combination of properties.

3. Over 400 F. (or maybe 450 F.) where the Mg-R.E. or Mg-R.E.-Mn casting alloys, with or without further alloying additions, exhibit the highest elevated temperature properties.

This division of service temperature ranges would allow the use of zirconium-containing alloys in the lower-elevated-temperature range and take advantage of their higher room-temperature properties, and better castability. For the highest temperature range, alloys of the type proposed in the present paper would be the obvious choice because of their superior creep characteristics.

Another problem is the heat treatment of these alloys. It is known from work on other magnesium-base alloys that for most of them the best creep characteristics at elevated temperatures are being obtained in the "as cast and stabilized" condition. The authors show all their results in the fully heat-treated condition, and it would be interesting to compare the difference in creep properties for these two conditions.

For the practical use of the proposed alloy it would be essential to answer the following questions:

1. What are the casting characteristics of the proposed alloy as compared with other magnesium casting alloys, especially the fluidity, shrinkage, tendency to segregation and hot cracking?

2. What are the welding characteristics of this alloy and how is welding affecting its properties?

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[1] Head, Non-Ferrous Metals Section, Canadian Bureau of Mines, Ottawa, Ontario, Canada.

[2] J. W. Meier and M. W. Martinson, "Magnesium Casting Alloys for Elevated Temperature Service," presented at Annual Meeting, Canadian Institute of Mining and Metallurgy, Toronto, April 18, 1950.

3. What effect has the nickel addition on the corrosion resistance of this alloy?

MR. R. T. PARKER.[3]—I should like to ask a question about the properties given in Table I on magnesium-cerium cast alloys, in which some deformation properties are given. Does the author consider it justifiable to compare the deformation properties of these alloys and the aluminum-base alloy by dividing by the density? I question the justification for doing so. It does bring the magnesium alloys a little nearer the aluminum alloys, but I should think it would have been more proper to consider the deformation properties on an absolute basis alone.

As an extension to this I wonder whether the author could give us a comparable value for the aluminum-6 per cent magnesium alloy and whether, in addition, he has any comparable properties for any of these alloys at room temperature?

MR. L. W. EASTWOOD (*author*).—Mr. Meier's classification of temperature service is well taken. The experimental alloy described in this paper is of course for service at temperatures in the range 500 to 600 F. All of the information on the foundry characteristics is described in the paper. No information is available on its welding characteristics. The magnesium-cerium alloys will require paint protection when used in corrosive atmospheres. The data presented in the paper indicate that heat treating the magnesium-cerium alloy of the EM42 type has little or no effect on the creep properties.

In reply to Mr. Parker's question, it seems quite reasonable in aircraft castings to compare materials on an equal weight basis. This is the reason light alloys are so extensively used in aircraft. On an equal weight basis, the high-temperature properties of the magnesium-cerium alloys are better than those of the aluminum-6 per cent, magnesium alloy described elsewhere.[4]

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[3] Director of Research Dept., Aluminum Laboratories Limited, Inc., Banbury, Oxon, England.

[4] L. W. Eastwood, Webster Hodge, and C. H. Lorig, "Aluminum—6 per cent Magnesium Alloys for Elevated-Temperature Service," see p. 1013 of this publication.

ALUMINUM - 6 PER CENT MAGNESIUM ALLOYS FOR ELEVATED-TEMPERATURE SERVICE*[1]

By L. W. Eastwood,[2] Webster Hodge,[2] and C. H. Lorig[2]

Synopsis

As a result of a systematic study of aluminum-base alloys containing magnesium, the following casting alloy has been developed for elevated-temperature service:

Magnesium	6 per cent	Zirconium	0.05–0.25 per cent
Manganese	1 per cent	Titanium	0.08 per cent
Copper	1.5 per cent	Beryllium	0.005 per cent
Vanadium	0.1 per cent	Aluminum (99.5 per cent)	Balance

This alloy is not appreciably adversely affected by stabilization at temperatures up to 700 F., has low density, good resistance to corrosion, good machining characteristics, and, even after stabilization at 600 F., has very high tensile properties. As compared with 142 or Y alloy castings, it has appreciably lower thermal conductivity and somewhat lower resistance to creep at 600 F. Although the above composition has considerably better resistance to creep than previously known aluminum-6 per cent magnesium casting alloys, still further improvements in creep resistance may be possible.

Aluminum-base alloys have had wide application in aircraft motor parts operating at elevated temperatures. It is known that, in some instances, such operating temperatures are as high as 600 F. Among the aluminum-base casting-alloys, 142 alloy (4 per cent copper–1.5 per cent magnesium–2 per cent nickel) and A355 (1.4 per cent copper –5 per cent silicon–0.5 per cent magnesium–0.8 per cent nickel–0.8 per cent manganese) are most commonly recommended for sand-cast parts for elevated-temperature service.

It is well known that the aluminum-base alloys containing 6 per cent magnesium have higher tensile properties at 600 F. than either 142 or A355 compositions.[3, 4] Such magnesium-containing alloys are also outstanding because of their excellent tensile properties at room temperature, good machining characteristics, excellent resistance to corrosion, and low density. Although the aluminum-base alloys containing magnesium do not crack in the mold during cooling after solidification, it is necessary to use good gating practice to avoid defects resulting from turbulence during the pour. Some precautions against reaction with the sand are also required. Usually, in an aluminum alloy containing as much as

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[*] Presented at the Fifty-third Annual Meeting of the Society, June 26-30, 1950.

[1] The work described in this paper was sponsored and financed by the National Advisory Committee for Aeronautics, Contracts Nos. NAw-5480 and NAw-5627. Part of this work has been described in *NACA Technical Note No. 1444*, issued January, 1948.

[2] Supervisor, Assistant Supervisor, and Assistant Director, respectively, Battelle Memorial Inst., Columbus, Ohio.

[3] Alcoa Aluminum and Its Alloys, Aluminum Company of America, 1947.

[4] L. W. Kemp, W. A. Dean, and R. B. Wrege, "Investigation of Aluminum Alloys for Applications at Elevated Temperatures," *Aloca Report No.* 4-45-6, Cleveland Research Division, Aluminum Company of America, September 12, 1945.

6 per cent magnesium, sand reaction can be avoided, at least in the light sections, by adding 0.005 per cent beryllium to the alloy.

For high-temperature applications, the aluminum alloys containing 6 per cent magnesium have two principal disadvantages. One of these is low thermal conductivity which, after heat treating and stabilizing the casting, is about one-third lower than that of 142 in a heat-treated and stabilized condition. The other disadvantage of the aluminum-6 per cent magnesium alloys for elevated-temperature applications is their poor resistance to creep[5] at 500 and 600 F., despite their relatively high tensile strength and yield strength at these temperatures.

For those applications in which heat dissipation is unimportant and service life short, it was believed that aluminum-base alloys containing 6 per cent magnesium would be very useful. In such applications where the service life at temperature is short, high tensile properties would be desirable, but resistance to creep would not be important. It was thought that a modification might be made in the composition which would improve the resistance to creep and, therefore, make alloys of this type useful for high-temperature applications involving prolonged service life. Accordingly, the principal objective of the experimental work described in this paper was the development of improved creep properties in the aluminum-6 per cent magnesium-base alloys at 600 F. Some attention was paid to the improvement of tensile properties at room temperature to 600 F.

EXPERIMENTAL WORK

The general procedure consisted of casting the experimental compositions into test bars made in green sand molds. A standard four-bar mold was used for this purpose. The test bars poured from 1320 to 1350 F. were usually solution-heat-treated and aged or stabilized, but in some instances, they were tested in the as-cast as well as as-cast and stabilized conditions to determine the effects of heat treatment. Metallographic examination was made of each experimental composition to make certain that the test bars were sound and fine grained. Those compositions having good room-temperature tensile properties were also subjected to tension tests at 600 F. and to a standard creep test at 600 F., using a 1300 psi. load.

Sand Reaction:

One of the first difficulties encountered in the early development work with the 6 per cent magnesium alloys was the tendency of the alloy to react with moisture in the green sand molds. The seriousness of this sand reaction varied from mold to mold, but, in general, the surface unsoundness which resulted would entirely vitiate any useful comparison of the properties of the sand-cast test bars of various compositions.

It was found that approximately 0.005 per cent beryllium, when added to the 6 per cent magnesium melt, would entirely eliminate this trouble from sand reaction. Although the amount of beryllium required is not critical, it should not be much in excess of 0.005 per cent; otherwise, high beryllium content will produce a characteristic sand reaction with the green sand molds. These beryllium additions were made to all the melts by adding an aluminum-beryllium alloy containing 1 to 1.25 per cent beryllium. When scrap was melted, no additional beryllium was required. While the beryllium addition was very effective, it should be noted that it may not be adequately effective when casting heavy sections. In this case, an inhibitor in the sand will be required.

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[5] R. R. Kennedy, "Creep Characteristics of Aluminum Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, pp. 218–231 (1935).

Grain Refinement:

In order to avoid variations in grain size from one composition to another, a grain refiner was added to all the experimental compositions. It was found that titanium was very effective, and an addition of either 0.08 per cent titanium alone or the combination of 0.01 per cent boron plus 0.02 per cent titanium was used for this purpose. Either of these additions resulted in consistently fine grain, usually of the order of 0.01 to 0.02 in. in the test bars poured from 1325 to 1350 F.

Melt Quality:

The 6 per cent magnesium alloys, like other alloys of aluminum which solidify essentially as a solid solution, are subject to an unsoundness called microporosity. In well-fed castings such as the test bars, such unsoundness is caused by the evolution of gas (hydrogen) during solidification. Microporosity has a very marked effect upon the tensile properties of the castings. Therefore, to permit accurate comparison of one composition with another, the production of consistently sound test bars was insured by chlorine fluxing the melts at 1325 to 1350 F. for 15 min. just prior to removing the pot and pouring the melt.

Heat Treatment:

Although aluminum alloys containing 6 per cent magnesium are not normally considered to be amenable to heat treatment, it was found that some of the compositions were very markedly improved by solution heat treatment. This was especially true of the aluminum-6 per cent magnesium alloys containing 1.5 per cent copper. Furthermore, these benefits of the solution heat treatment were retained in the alloy after it was exposed for long periods at temperatures of 650 F. Thus, alloys of this type are structurally stable at service temperatures of the order of 600 to 650 F. This is in very marked contrast to the effects of exposure to high temperature on the room-temperature and elevated-temperature properties of 142 alloy. Since the anticipated service conditions would involve prolonged exposure to a temperature of about 600 F., all of the experimental compositions were stabilized for 24 hr. at 650 F. and then air cooled from the stabilization temperature. To establish the effects of heat treatment, some of the alloys were tested in the as-cast or as-cast and stabilized conditions. When a heat treatment was employed, the castings containing 6 per cent magnesium were heated for 16 hr. at 810 F., quenched in cold water, and then stabilized for 24 hr. at 650 F. before testing them at room temperature or at 600 F. This is referred to as the HTS condition. The effect of heat treating an alloy containing 6 per cent magnesium, 1.5 per cent copper, 1 per cent manganese, 0.02 per cent titanium, 0.01 per cent boron, 0.005 per cent beryllium, balance 99.75 per cent aluminum, is shown by the following data:

	As Cast	Heat Treated and Stabilized
Tensile strength, psi.	26 500	35 000
Yield strength, 0.2 per cent offset, psi.	26 000	26 000
Elongation in 2 in., per cent	0.8	2.7

The effects of prolonged exposure of this alloy, 142 alloy, and Alcoa 254 alloy at 650 F. are shown by the data in Table I. It is quite evident from this table that the room-temperature tensile properties of the two alloys containing 6 per cent magnesium are not appreciably damaged by prolonged exposure at 650 F. On the other hand, 142 or Y alloy is quite adversely affected by such a treatment. The yield strength of 142 alloy after exposure for 18 days at 650 F. is only about 40 per cent of that of the aluminum - 6 per cent magnesium alloys similarly stabilized.

Tension and Creep Tests:

Because the anticipated service temperature was 600 F. and the service life a prolonged period, the alloys were tested at 600 F. in the stabilized condition. When carrying out the tension tests at 600 F., the bars previously stabilized were held at temperature ½ hr. before making the test. The rate of movement of the crosshead was 0.01 in. per min. per in. of gage length until the 0.2 per cent offset yield was passed. The rate of the crosshead movement was then increased to 0.03 in. per min. per in. of gage length until rupture was produced.

TABLE I.—EFFECT OF STABILIZATION ON THE ROOM TEMPERATURE PROPERTIES OF THREE ALLOYS[a]

	Additional Stabilization Treatment	Alcoa 142 HTA[b]	Alcoa 254 As Cast	Experimental Alloy HTS[b]
Tensile strength, psi	None	39 500	24 000	35 000
Yield strength, 0.2 per cent offset, psi	None	37 000	23 000	24 500
Elongation in 2 in., per cent	None	1.5	1.0	2.6
Tensile strength, psi	18 days at 650 F.	24 000	28 000	30 500
Yield strength, 0.2 per cent offset, psi	18 days at 650 F.	9 600	22 700	22 700
Elongation in 2 in., per cent	18 days at 650 F.	2.5	1.0	1.7

[a] Alcoa 142 initially heat treated 6 hr. at 960 F., quenched in boiling water, aged 8 hr. at 400 F. Alcoa 254 was in the as-cast condition. Experimental alloy was heat treated 16 hr. at 810 F., quenched in cold water, stabilized 24 hr. at 650 F., air cooled. The nominal compositions of the alloys, in per cent, were as follows:

	Copper	Manganese	Nickel	Magnesium	Titanium	Boron	Balance
Alcoa 142	4.0	...	2.0	1.5	0.1	...	99.5 per cent Al
Alcoa 254	0.5	1.0	1.5	6.0	0.02	0.01	99.75 per cent Al
Experimental[c]	1.5	1.0	...	6.0	0.02	0.01	99.75 per cent Al

[b] HTA = Heat treated, aged.
HTS = Heat treated, stabilized.

[c] The experimental alloy is not the alloy of optimum composition. The latter alloy would have slightly better tensile properties.

The creep tests were conducted at 600 F., employing a load of 1300 psi. This creep test was used as a method of screening the alloys to indicate those having the greatest resistance to creep. The test was run for approximately one week. When obtaining creep data on the alloy of optimum composition, the tests were run for longer periods.

A detailed account of the method of making the tension tests and creep tests at elevated temperature has been described elsewhere.[6]

Compositions Investigated:

A considerable number of experimental alloys were prepared, most of them containing 6 per cent magnesium. The range in compositions investigated is shown by the data in Table II. As indicated previously, all of these alloys contained 0.005 per cent beryllium and either 0.08 per cent titanium or 0.01 per cent boron and 0.02 per cent titanium. The beryllium was added to prevent sand reaction, and the grain refiner was added to produce fine-grain castings. As a result of these studies, the effects of the principal alloy elements were established as described in the following paragraphs.

Effect of Magnesium Content.—Between the limits of 4 per cent and 6 per cent magnesium, very little difference was noted in the tensile strength of the experimental alloys at 600 F. In the heat-treated condition, the room-temperature properties, of course, increase up to about 11 per cent magnesium. However, in the heat-treated and stabilized condition, 6 per cent magnesium is the optimum for maximum room-temperature tensile properties. At concentrations above 6 per cent magnesium, the

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[6] K. Grube and L. W. Eastwood, "Magnesium-Cerium Cast Alloys for Elevated-Temperature Service," *Proceedings*, Am. Soc. Testing Mats., Vol. 50, p. 989 (1950).

TABLE II.—RANGE OF ALLOY COMPOSITIONS INVESTIGATED.

Base Composition, per cent						Additions	Range
Copper	Manganese	Nickel	Iron	Magnesium	Other		
...	...	...	...	6	...	None	...
						Ce	0.0 to 5.0
						Mn	0.05 to 1.5
						Co	0.0 to 0.75
						Cu	0.0 to 13.0
						Sb	0.0 to 2.0
						Ni	0.0 to 2.5
						Zn	0.0 to 5.0
...	1	...	...		...	Mg	2.0 to 10.0
...	1	...	...	6	...	Ti	0.0 to 0.2
						Cu	0.0 to 3.0
						Ni	0.0 to 3.0
						Ag	0.5 to 4.0
...	...	2	...	6	...	None	...
						Fe	0.0 to 1.0
...	...	...	1	6	...	Ni	0.5 to 2.0
...	...	1.5	1	6	...	Mn	0.0 to 0.75
...	1	2	1	6	...	None	...
						Zr	0.1
...	...	...	...	5	...	Fe	0.32 to 4.11
...	...	...	2.5	5	...	Ni	0.0 to 4.0
...	1	1	...	6	...	None	...
						Cu	0.0 to 2.0
						Fe	1.0
						Co	0.0 to 0.75
						Zr	0.0 to 0.5
						Ce	0.0 to 1.0
						Sb	0.0 to 1.0
						Cr	0.0 to 0.5
						W	0.0 to 1.0
						Mo	0.0 to 1.0
...	1	1	...	6	0.25 Zr	V	0.1 to 0.4
...	1	1	...	6	0.5 Sb	Zr	0.1
						W	0.1
						Mo	0.1
						Ca	0.1
...	1	1	...	6	0.1 Zr	Cu	0.1
1.5	1	1.5	...	6	...	None	
						Cr	0.0 to 0.5
						Co	0.0 to 1.0
						Sb	0.0 to 0.5
						Ti	0.0 to 0.4
1.5	...	0.5	...	6	...	Ag	0.25 to 2.0
						Zn	0.0 to 1.0
						Si	0.0 to 1.0
						Co	0.5
						Sb	0.5
						Ce	0.5
1.5	...	0.5	...	5	...	Zn	0.0 to 2.0
1.5	1.0	...	...	6	...	Ni	0.0 to 2.5
						Cr	0.0 to 0.5
						Co	0.0 to 1.0
						Ce	0.0 to 3.0
						Sb	0.0 to 0.5
						Mo	0.0 to 0.5
						Zr	0.0 to 0.5
						Ca	0.0 to 0.5
						Cd	0.0 to 2.0
						Li	0.0 to 1.0
1.5	1.0	...	...	6	0.25 Zr	V	0.0 to 0.4
						Cr	0.0 to 0.1
1.5	1.0	...	...	6	0.1 Zr	Sb	0.0 to 0.5
						W	0.0 to 0.1
						Mo	0.0 to 0.1
						Cu	0.0 to 0.1
						Ti	0.0 to 0.15
						V, Ca, Co, Ce	0.05 each
1.5	1.0	...	...	6	0.05 Ca	Li	0.05

tensile strength at 600 F. decreases rapidly, and the precipitated aluminum-magnesium compound can be noted at the grain boundaries.

Some stabilization tests were carried out with alloys containing 4, 5, and 6 per cent magnesium, the alloys otherwise being similar because all contained 1.5 per cent copper, 1 per cent magnanese, with grain refiner and beryllium additions. The effects of stabilization at 1, 4, and 10 days at 575 and 650 F. were determined after solution heat treatment. The alloy with 4 per cent magnesium showed no appreciable change in the room-temperature properties with increasing time at either of the stabilization temperatures. The 5 per cent magnesium alloy showed a slight gain in ultimate strength at room temperature after all stabilizing treatments extending beyond the 24-hr. period, but the yield strength was not affected. On the other hand, the 6 per cent magnesium alloys showed a slight decrease in ultimate strength and hardness after all stabilizing treatments which were prolonged beyond the 24-hr. period. This adverse effect of prolonged heating at 575 or 650 F. was slight, and it indicates that, in general, these alloys containing up to 6 per cent magnesium are structurally stable in this temperature range. Magnesium contents appreciably higher than 6 per cent are not desirable.

Effect of Manganese Content.—Manganese up to 1.5 per cent can be tolerated in the 6 per cent mangesium alloys, and the optimum content of approximately 1 per cent is a very desirable addition because it improves the tensile properties at room temperature and at 600 F. However, an aluminum alloy containing 6 per cent magnesium and 1 per cent manganese has rather poor ductility at 600 F. because of the large amount of cracking which occurs under tension, with relatively little elongation.

Effect of Copper Content.—Copper

added to an aluminum-base alloy containing 6 per cent magnesium and 1 per cent manganese improves the ductility both at room temperature and at high temperature, particularly because the cracking which occurs in tension bars broken at 600 F. is substantially eliminated by the copper addition. In place of the numerous small cracks, uniform elongation and considerable necking at the fracture occur. The room-temperature tensile properties of the alloy containing the copper are also subject to marked improvement by a solution heat treatment, followed by an aging or stabilizing treatment consisting of 24 hr. at 650 F. As a result, the optimum copper addition of 1.5 per cent produces a marked improvement in the room-temperature tensile properties of the solution heat-treated and stabilized alloy.

Effect of Nickel Content.—Nickel may be substituted for copper either alone or combined with iron. About 2 per cent nickel with 1 per cent iron, or 1 per cent nickel with normal iron content as an impurity, appears to be the optimum composition for the aluminum-magnesium-nickel alloys. The room-temperature tensile properties of these alloys are moderately low and are not improved by heat treatment, but they have high tensile properties at 600 F.

Effect of Copper and Nickel Content.—Copper and nickel together in most proportions appear to be inferior to either alone. The best proportions of copper and nickel were found to be either 1.5 per cent nickel and 0.5 per cent copper, corresponding to Alcoa 254, or the reverse of these proportions. Both of these alloys, containing either 1.5 per cent nickel and 0.5 per cent copper, or 0.5 per cent nickel and 1.5 per cent copper, can be improved somewhat by a solution heat treatment, but to a much lesser extent than the nickel-free experimental alloy containing 1.5 per cent copper.

Optimum Compositions:

On the basis of the tensile properties of the various compositions at room temperature and at 600 F., the following 5 alloys, prepared from 99.7 per cent aluminum, were selected for further consideration:

Alloy	Composition, per cent						
	Magnesium	Manganese	Copper	Nickel	Titanium	Beryllium	Iron
No. 1..	6	1	1.5	0	0.08	0.005	...
No. 2..	6	1	...	1	0.08	0.005	...
No. 3..	6	1	...	2	0.08	0.005	1.0
No. 4..	6	1	1.5	0.5	0.08	0.005	...
No. 5..	6	1	0.5	1.5	0.08	0.005	...

The first of these was considered to have the greatest possibilities. After a solution heat treatment and a stabilization treatment consisting of 24 hr. at 650 F., this alloy consistently possessed the following mechanical properties:

	Room Temperature	600 F.
Tensile strength, psi.	35 000	13 000
Yield strength, psi.	24 500	10 000
Elongation in 2 in., per cent	2.5	20
Brinell hardness number	80	...

After complete stabilization or, at least, after 480 hr. at 650 F. prior to testing at 600 F., the tensile and yield strengths obtained at 600 F. did not decrease more than 1000 psi. from the above values. As indicated later, substantial improvements have been made in this composition by suitable additions.

The second alloy did not respond to solution heat treatment and, as a result, the room-temperature tensile properties were substantially lower than those obtained for Alloy No. 1, but the tensile properties at 600 F. were slightly superior to those obtained on the unimproved No. 1 composition.

The third alloy had relatively poor

yield strength at room temperature, but, nevertheless, had slightly the highest yield strength at 600 F. This composition did not respond to solution heat treatment, and, as a result, the room-temperature tensile properties were relatively low.

Composition No. 4 is similar to Alcoa 254, excepting that 0.08 per cent titanium has been added to produce grain refinement, and beryllium has been added to eliminate sand reaction, thereby improving the foundry characteristics when using green sand molds.

In general, then, the first of the five alloys listed has the best room-temperature properties, and at all temperatures up to and including 500 F. The other four alloys have tensile properties slightly lower than those of Alloy No. 1 at all temperatures up to 500 F., and all five alloys have similar tensile properties at 600 F. However, Alloy No. 3 has slightly the lowest room-temperature properties and slightly the highest tensile properties at 600 F.

Improvement in Resistance to Creep at Elevated Temperatures:

The existing information and the data obtained in the short-time creep tests definitely indicate that the aluminum-6 per cent magnesium alloys have poor resistance to creep at 600 F. Of the five compositions listed previously, which appeared to have some promise, Alloys Nos. 1 and 2 were subjected to further development in order to improve their tensile properties at room temperature and at 600 F., as well as to improve their resistance to creep at 600 F. Accordingly, the effects of many minor elements and combinations of minor elements were investigated in an effort to improve the creep properties.

The short-time creep tests were made on experimental alloy compositions, and on Alcoa 142 and Alcoa 254 for purposes of comparison. These short-time tests were carried out at 600 F., using a load of 1300 psi. for periods generally up to 100 to 150 hr., though some tests were continued for longer periods. It was, of course, necessary also to obtain tensile properties at room temperature and at 600 F. The entire series of experimental compositions prepared for creep testing is included in Table III. This table shows intended composition of the alloy, the tensile properties at room temperature, the maximum grain size, the tensile properties at 600 F., and some data on the creep properties at 600 F. employing a load of 1300 psi. A number of elements were found to have beneficial effects on the creep properties of the two base compositions.

Typical time-deformation curves for the creep tests run at 600 F. and 1300 psi. are shown by Fig. 1. This chart graphically shows the relative merits of Alcoa 142, Alcoa 254, and an experimental alloy. This graph also shows the beneficial effects on the creep rate obtained by the use of various amounts of zirconium in a base alloy containing 6 per cent magnesium, 1.5 per cent copper, 1 per cent manganese, with the usual amounts of grain refiner and beryllium. Figure 2 is a graphical representation of the more important creep data listed in Table III. This chart shows the minimum creep rate and the total deformation at 100 hr. obtained on alloys of various compositions.

Zirconium in amounts from 0.05 to 0.25 per cent increases the resistance of the base alloy to creep and tends to increase the room-temperature strength unless the grain size also increases. If a grain-size increase occurs at 0.02 per cent titanium addition, grain refinement may often be restored by increasing the titanium content to 0.08 per cent. Vanadium additions of 0.1 per cent in combination with 0.25 per cent zirconium

TABLE III.—PROPERTIES OF SOME EXPERIMENTAL SAND CAST ALUMINUM ALLOYS.

Heat Number	Heat Treatment[a]	Intended Composition, per cent								Properties at Room Temperature					Properties at 600 F.							
		Magnesium	Copper	Nickel	Manganese	Titanium	Boron	Beryllium	Other	Tensile Strength, psi.	Yield Strength, psi.	Elongation, per cent	Brinell Hardness Number	Grain Size, in.	Tensile[b] Strength, psi.	Yield[c] Strength, psi.	Elongation, per cent	Reduction of Area, per cent	Creep Tests at 1300 psi.			
																			Duration, hr.	Minimum Creep Rate, per cent per hr..	Total Deformation at 100 hr., per cent	Total Deformation at Completion of Test, per cent
76	HTA	1.5	4.0	2.0	...	0.10	...	...	...	42 500	41 200	1.1	111	0.020					139[e]	0.00010	0.045	0.051
76	HTAS	1.5	4.0	2.0	...	0.10	...	...	...	26 350	11 410	3.2	60	0.020	11 450	7 570	21.5	31.0	142	0.00018	0.042	0.050
185	HTA	1.5	4.0	2.0	...	0.10	...	...	...	41 220	41 200	0.6	117	0.015								
185	HTAS	1.5	4.0	2.0	...	0.10	...	...	...	24 970	13 920	2.1	62	0.015	12 030	9 050	12.0	9.2				
74	ac A	6.0	0.5	1.5	1.0	0.02	0.01	0.005	...	25 600	25 500	0.8	84	0.010								
74	ac AS	6.0	0.5	1.5	1.0	0.02	0.01	0.005	...	26 600	21 700	1.6	83	0.010	14 480	11 550	20.0	26.0	186	0.00075	0.123	0.165
118	HTS	4.0	1.5	0.5	1.0	0.02	0.01	0.005	...	26 300	19 930	1.2	78	0.010	13 250	11 440	58.0	50.0	144	0.00023	0.048	0.062
119	HTS	5.0	1.5	0.5	1.0	0.02	0.01	0.005	...	26 850	22 680	1.1	80	0.010	14 550	12 460	24.5	23.0	143	0.00033	0.054	0.066
120	HTS	6.0	1.5	0.5	1.0	0.02	0.01	0.005	...	31 125	23 180	1.7	84	0.010	13 120	10 290	25.0	24.0	142	0.00040	0.072	0.089
129A	HTS	6.0	1.5	0.5	1.0	0.02	0.01	0.005	0.5 Co	29 150	[illegible]	1.5	80	0.010	14 000	12 325	18.5	23.0	141	0.00025	0.048	0.054
130A	HTS	6.0	1.5	0.5	1.0	0.02	0.01	0.005	0.5 Sb	31 100	21 500	2.3	73	0.05 to 0.015	13 310	11 340	68.5	49.5	136	0.00013	0.050	0.052
131A	HTS	6.0	1.5	0.5	1.0	0.02	0.01	0.005	0.5 Ce	23 900		0.5	74	0.005	12 750	10 210	19.5	19.0	578	0.000075[f]	0.066	0.105
78A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	...	35 300	24 550	2.6	84	0.015	13 350	10 800	50.5	50.0	164[e] 143	0.00045 0.00040	0.074 0.053	0.107 0.075
99A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.1 Zr	38 000	...	3.1	83	0.015	12 790	10 250	60.0	60.0	140	0.00015	0.060	0.068
112A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.1 Zr	36 310	25 050	2.7	83	0.03 to 0.05	13 390	10 210	52.5	53.0	504	0.000080[g]	0.030	0.066
98A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.2 Zr	33 600	...	2.3	76	0.01 to 0.03	12 660	9 960	23.0	20.0	118	0.00027	0.073	0.079
111A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.2 Zr	37 500	...	2.6	80	0.020	14 660	11 640	56.5	54.0	144	0.00013	0.032	0.039
145A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	...	33 100	...	1.7	82	0.010	...	...	...	...	...	...	...	...
148A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.05 Zr	37 680	...	2.7	83	0.010	13 250	11 000	47.5	50.5	146	0.00016	0.050	0.059
145B	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr	39 850	...	3.4	82	0.010	14 020	11 160	58.0	50.5	168	0.00033	0.071	0.087
145C	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.25 Zr	36 500	...	2.7	83	0.010	13 560	10 840	59.5	45.0	144	0.00025	0.040	0.051
145D	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.5 Zr	38 400	...	2.2	83	0.007	13 460	11 300	40.0	34.0	144	0.00085	0.140	0.176
149A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.25 Zr 0.1 V	38 720	25 300	3.3	87	0.020	13 440	10 620	71.0	58.5	482	0.000035[h]	0.045	0.078
160A[d]	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.25 Zr 0.25 V	33 550	...	1.9	83	0.050	13 610	11 070	34.0	30.0	169	0.00006	0.031	0.034
149B	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.25 Zr 0.4 V	32 420	24 820	2.1	81	0.015	14 800	10 860	65.5	57.5	145	0.00010	0.035	0.039
150A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.25 Zr 0.1 Cr	32 470	24 920	2.2	78	0.015	14 500	11 610	30.5	41.0	167	0.00010	0.042	0.052
167A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr 0.5 Sb	30 450	...	2.0	79.6	0.010	13 610	11 510	77.0	62.2	287	0.00006	0.040	0.059
168A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr 0.1 V	37 300	...	2.7	83	0.020	12 970	10 370	>102.0	>92.0	219	0.00005	0.030	0.037
169A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr 0.1 W	40 100	...	3.7	85	0.008	13 300	10 820	95.5	69.0	143	0.00027	0.078	0.087
170A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr 0.1 Mo	33 350	...	2.3	84	0.010	13 700	11 500	88.0	65.5	140	0.00018	0.062	0.070
172A	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr 0.1 Ca	38 500	...	3.1	84	0.010	13 510	11 500	31.0	28.0	140	0.00018	0.052	0.058
173A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.1 Zr	38 550	...	4.0	81	0.02 to 0.03	12 860	10 880	79.0	66.5	140	0.00008	0.029	0.033
173B	HTS	6.0	1.5	...	1.0	0.08	0.01	0.005	0.1 Zr	37 200	...	3.7	80	0.010	13 220	11 360	67.0	61.0	...	...	...	...
173C	HTS	6.0	1.5	...	1.0	0.15	0.01	0.005	0.1 Zr	37 070	...	3.8	78	0.008	13 150	11 320	36.0	33.0	...	...	...	...
139A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.003 Ca	36 100	...	2.7	86	0.015	12 800	12 100	60.0	60.0	166	0.00025	0.057	0.080
138A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.03 Ca	38 630	...	2.9	82	0.015	13 500	10 670	53.0	51.5	136	0.00023	0.049	0.057
137A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.06 Ca	38 800	...	2.9	79	0.015	12 710	10 650	67.5	54.5	211	0.00022	0.071	0.094
136A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.12 Ca	38 540	23 600	3.5	84	0.03 to 0.04	13 140	11 000	53.0	43.5	500	0.00007[i]	0.035	0.065

135A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.25 Ca		35 600	...	2.7	85	0.020	12 690	11 550	44.0	42.0	503	0.00021[j]	0.035	0.121
134A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.5 Ca		28 080	...	1.5	76	0.03 to 0.04	...	...	...	...	...	...	...	...
179A	HTS	6.0	1.5	...	1.0	0.02	0.01	0.005	0.1 Ca	0.1 V	32 690	...	3.0	76	0.020	13 140	11 320	62.0	63.5	...	...	...	...
180A	HTS	6.0	1.5		1.0	0.02	0.01	0.005	0.1 Zr 0.05 V 0.05 Co	0.05 Ca 0.05 Ce	33 050		2.5	81	0.050	13 510	11 750	71.0	90.0				
86A	HTS	6.0		1.0	1.0	0.20	...	0.005			32 100		2.7	78	0.010	12 150	10 170	13.5	16.0	123	0.00053	0.065	0.075
87A	HTS	6.0	1.5	1.0	1.0	0.20	...	0.005			33 600		1.3	82	0.010	12 100	9 580	15.0	17.0	162	0.00040	0.093	0.116
89A	HTS	6.0		1.0	1.0	0.20	0.01	0.005	1.0 Fe		29 815		1.9	77	0.015	12 200	9 390	13.0	11.0	141	0.00050	0.110	0.130
104A	HTS	6.0		1.0	1.0	0.01	0.01	0.005	0.1 Zr		28 800		1.9	80	0.05 to 0.08	13 400	9 880	10.0	13.0	197	0.00015	0.053	0.066
103A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	0.2 Zr		31 000		3.2	75	0.020	12 400	8 440	13.5	7.0	216	0.00017	0.075	0.094
151A	HTS	6.0		1.0	1.0	0.08	0.01	0.005			26 970		1.8	78	0.010	13 400	10 300	11.5	10.0	...	...	...	...
152A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.05 Zr		28 010	23 470	2.0	80	0.020	13 040	10 860	11.0	11.0	168	0.00018	0.039	0.050
151B	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.1 Zr		27 200		2.0	79	0.010	13 060	10 320	10.5	12.0	139	0.00037	0.108	0.120
151C	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.25 Zr		28 450		1.4	84	0.015	13 190	10 140	12.5	12.0	165	0.00017	0.053	0.066
151D	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.5 Zr		31 700		1.9	80	0.010	12 460	10 200	12.5	13.5	200	0.00030	0.090	0.119
153A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.25 Zr	0.1 V	27 700		1.7	81	0.030	13 040	11 240	11.0	9.5	526	0.000080[k]	0.030	0.065
154A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.25 Zr	0.25 V	27 620		2.0	83	0.050	12 610	10 670	9.0	6.5	142	0.00015	0.050	0.054
177A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	0.1 Zr	0.1 Ca	26 470		1.9	81	0.030	13 470	12 300	18.0	19.0				
109A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	0.25 Cr		27 500		1.5	82	0.010	12 860	10 370	10.0	9.5	163	0.00022	0.078	0.098
108A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	0.5 Cr		27 100		1.9	78	0.01 to 0.03	12 860	10 370	10.0	9.5	145	0.00020	0.062	0.071
158A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	0.12 W		29 400		2.0	81	0.050	13 140	10 670	10.0	8.0	220	0.00015	0.063	0.082
155A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	1.0 W		27 650		2.4	89	0.010	13 190	11 870	9.0	10.5	171	0.00030	0.070	0.090
163A	HTS	6.0		1.0	1.0	0.02	0.01	0.005	0.12 Mo		30 850		1.4	82	0.012	13 000	11 600	11.0	9.0	146	0.00025	0.060	0.073
164A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.5 Sb	0.1 Zr	30 150		1.5	78	0.012	12 970	11 300	13.5	12.5	145	0.00027	0.062	0.074
165A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.5 Sb	0.1 W	30 525		1.8	80	0.010	13 740	12 070	11.5	11.0	173	0.00020	0.047	0.061
166A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.5 Sb	0.1 Mo	28 450		2.0	80	0.015	13 560	11 700	17.0	18.0			0.038	
178A	HTS	6.0		1.0	1.0	0.08	0.01	0.005	0.5 Sb	0.1 Ca	29 140		1.7	76	0.010	13 540	11 670	9.5	9.5			0.040	
102A	HTS	6.0		1.5	1.0	0.02	0.01	0.005	1.1 Fe		26 450		1.6	80	0.02 to 0.03	14 400	>10 000	6.5	7.6	146	0.00050	0.119	0.136
110A	HTS	5.7		1.9	1.0	0.02	0.01	0.005	1.0 Fe		29 900		1.4	84	0.008	14 125	11 985	13.0	15.0	162	0.00020	0.058	0.070
181A	HTS	6.0		2.0	1.0	0.02	0.01	0.005	1.0 Fe	0.1 Zr	26 910	19 700	1.7	80	0.012	13 640	12 110	11.5	11.0	155	0.00018	0.053	0.061
182A	HTS	6.0	0.5	1.5	1.0	0.02	0.01	0.005	0.1 Zr		26 570	20 570	1.7	76	0.01 to 0.04	12.790	10 600	15.0	18.0	170	0.00018	0.044	0.054

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[a] HTA—Heat treated 6 hr. at 960 F., quenched in boiling water, aged 8 hr. at 400 F.
HTAS—Same as HTA, plus 24 hr. at 650 F.
ac A —As-cast bars, aged 8 hr. at 400 F.
ac AS—Same as ac A, plus 24 hr. at 650 F.
HTS—Heat treated 16 hr. at 810 F., quenched in cold water, plus 24 hr. at 650 F.

[b] All bars pulled at 0.01 in. per in. of gage length per min. until 0.2 per cent offset was passed. Rate was then increased to 0.03 in. per in. of gage length per min. to rupture.

[c] Yield strength at 0.2 per cent offset.

[d] Melt entirely produced from scrap metal.

[e] Creep tests run in duplicate.

[f] Minimum creep rate up to 150 hr., 0.00020 per cent per hr.

[g] Minimum creep rate up to 150 hr., 0.00020 per cent per hr.

[h] Minimum creep rate up to 150 hr., 0.00005 per cent per hr.

[i] Minimum creep rate up to 150 hr., 0.00007 per cent per hr.

[j] Minimum creep rate up to 150 hr., 0.00025 per cent per hr.

[k] Minimum creep rate up to 150 hr., 0.00012 per cent per hr.

produce excellent room-temperature properties, good high-temperature properties, and improved resistance to creep. Vanadium in greater amounts appeared to have a favorable effect on the high-temperature tensile strength but decreased the room-temperature tensile strength somewhat.

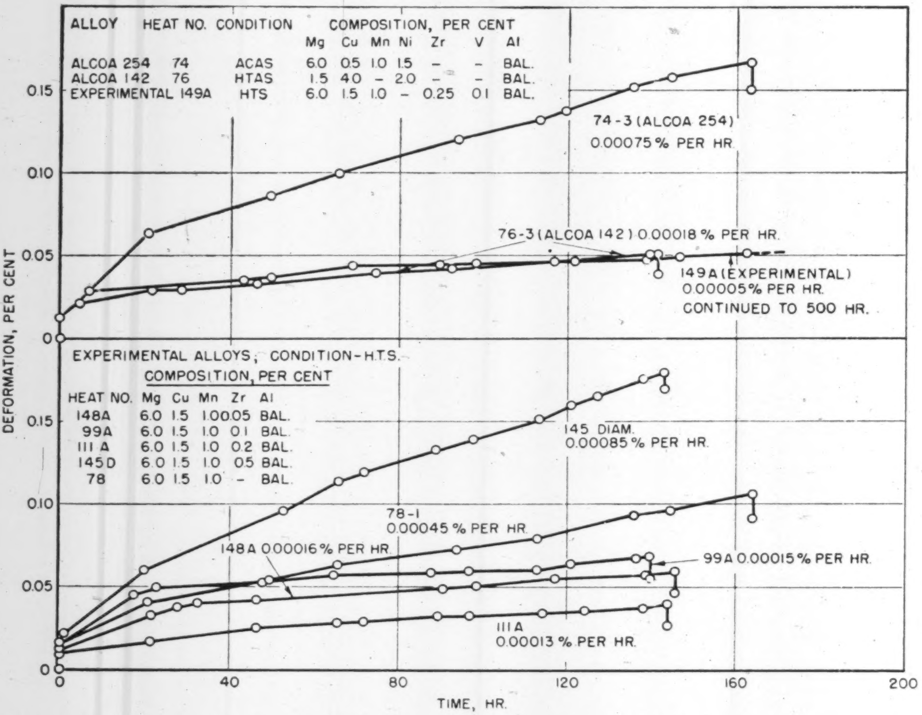

FIG. 1.—Typical Time-Deformation Curves for Creep Test Run at 600 F. and 1300 psi. on Cast Aluminum-Base Alloys

Tensile Properties of Stabilized Alloys:

During the course of the experimental work, speicimens of alloys for elevated-temperature service, developed by the Royal Aircraft Establishment (R.A.E.), were received for testing. Two alloys were available, each in the wrought and chill-cast conditions. Specimens of these four alloys in addition to Alcoa 142, Alcoa 254, and an experimental alloy containing 6 per cent magnesium, 1.5 per cent copper, and 1 per cent manganese, with grain refiners and beryllium, were stabilized as follows:

Tests	Stabilizing Treatment
Room temperature	None
400	480 hr. at 575 F.
500	480 hr. at 575 F.
600	480 hr. at 650 F.
700	96 hr. at 700 F.

Other specimens of the alloys were prepared and tested at room temperature in the following conditions:

Alloy	Condition
R.A.E. alloys	As received
Alcoa 142	Heat treated and aged
Alcoa 254	Aged 8 hr. at 400 F.
Experimental alloy	Heat treated and stabilized 24 hr. at 650 F.

The compositions of the various alloys are listed in Table IV. The tensile strength, yield strength, and elongation

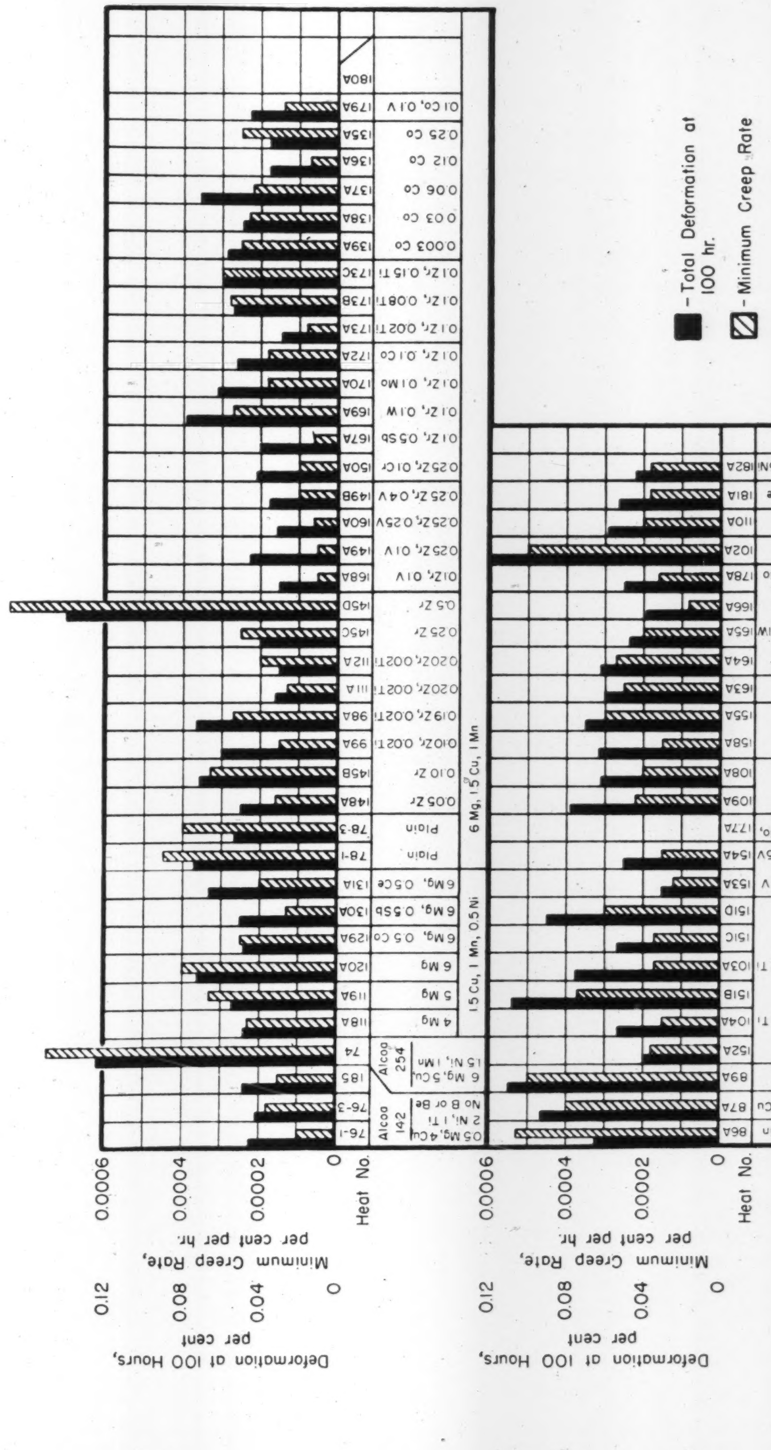

Fig. 2.—Summary of Data for Creep Tests Run at 600 F. and 1300 psi. on Cast Aluminum-Base Alloys.

Note: Unless otherwise stated all Alloys contain 0.01 per cent B, 0.005 per cent Be and either 0.02 per cent Ti (without Zr present) or 0.08 per cent Ti (with Zr present).

TABLE IV.—COMPOSITION OF STABILIZED ALLOYS REPRESENTED IN FIGS. 3, 4, AND 5.

Alloy	Composition, per cent										
	Copper	Manganese	Nickel	Magnesium	Chromium	Titanium	Boron	Beryllium	Silicon	Iron	Aluminum
R.A.E. 40 C.	2.30	1.87	5.02	0.68	0.51	0.07	...	0.34	0.18	0.32	Remainder
R.A.E. 55	1.75	1.75	2.85	0.73	0.50	0.05	...	...	0.13	0.20	Remainder
Alcoa 142[a]	4.00	...	2.00	1.50	...	0.10	...	...	0.10	0.41	Remainder
Alcoa 254[a]	0.50	1.05	1.50	5.99	...	0.02	0.01	0.005	0.09	0.16	Remainder
Experimental[a] (Heat 78)	1.50	1.05	...	6.00	...	0.02	0.01	0.005	0.09	0.14	Remainder
CM 62[a]	...	1.80	...	Remainder	6% cerium (rare earths)						...

[a] Calculated or nominal compositions.

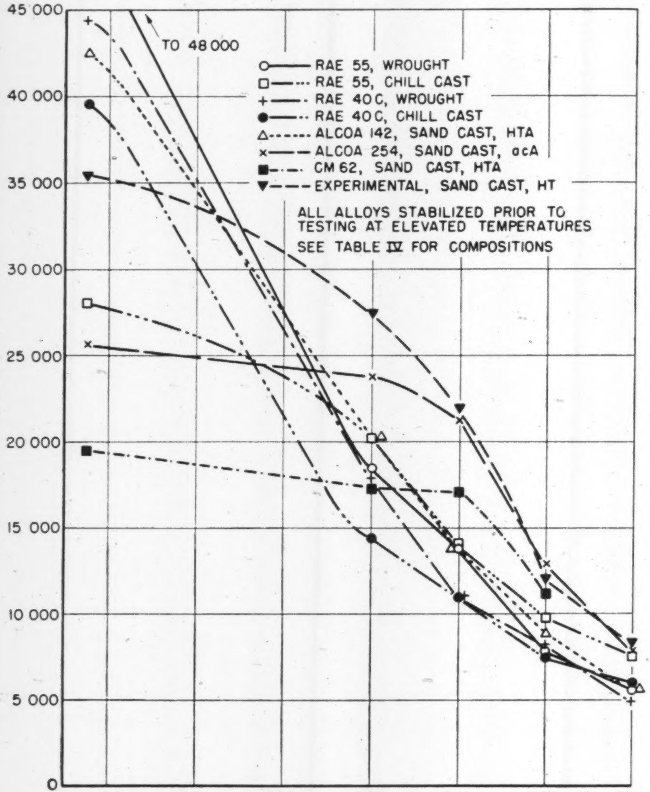

FIG. 3.—Tensile Strength of Various Alloys.

data obtained on these compositions are graphically illustrated by Figs 3, 4, and 5, respectively.

It should be noted that the experimental cast alloy compares favorably with the other two sand-cast compositions, Alcoa 142 and Alcoa 254, and even with the chill-cast R.A.E. alloy. The experimental 6 per cent mangesium alloy, however, is not the optimum composition

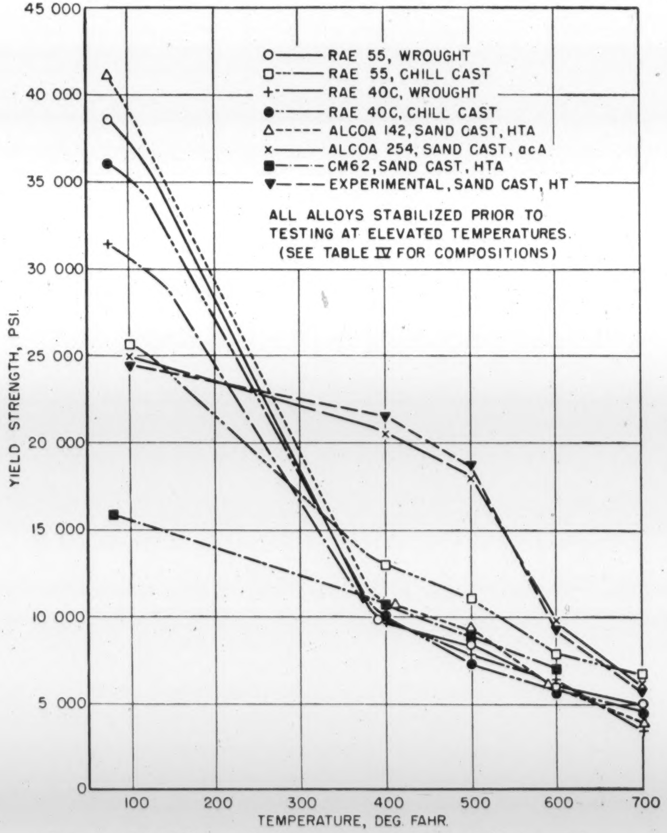

FIG. 4.—Yield Strength (0.2 per cent Offset) of Various Alloys.

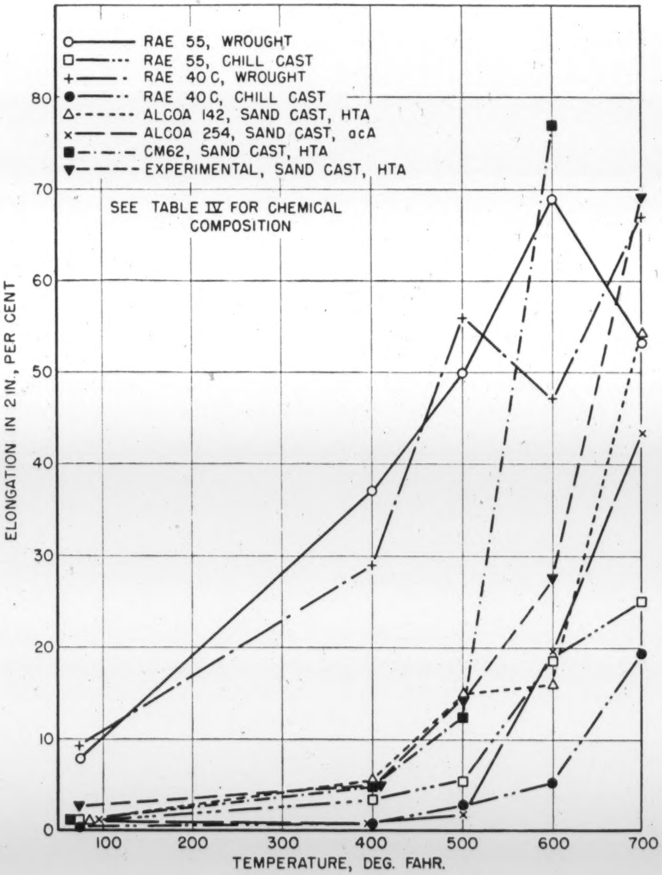

FIG. 5.—Elongation of Various Alloys.

which was later developed, because it did not contain zirconium or vanadium. If the experimental alloy had contained 0.1 per cent vanadium and 0.25 per cent zirconium, both the tensile properties at room temperature and at 600 F. would be slightly superior to those shown in Figs. 3, 4, and 5. It should be further noted that the R.A.E. alloys tested may not be representative of the best cast and wrought aluminum-base alloys developed in England for elevated-temperature service.

TABLE V.—CHEMICAL ANALYSIS OF THE ALLOYS LISTED IN TABLES VIII AND IX AND REPRESENTED BY FIGS. 6 TO 10.

Heat	Composition, per cent								
	Copper	Manganese	Magnesium	Titanium	Zerconium	Vanadium	Boron	Beryllium	Nickel
				6 PER CENT MAGNESIUM ALLOY					
A-3991	1.50	1.06	6.02	0.08	0.30	0.11	0.003	0.0005	....
A-3992	1.51	1.06	6.15	0.09	0.30	0.10	0.004	0.0005	....
A-4208	1.50	0.99	5.54	0.08	<0.05	0.12	0.005	0.005	....
A-4298	1.51	0.97	5.94	0.08	<0.05	0.13	0.004	0.005	....
				ALLOY NO. 142					
A-3993	3.87	....	1.43	0.09	......	....	....	......	1.89
A-3994	3.84	....	1.43	0.09	......	....	....	......	1.79
A-4299	4.01	0.02	1.52	0.08	......	....	....	......	2.27

Comparison of the Tensile and Creep Properties of 142 Alloy and the Experimental Alloy of Optimum Composition:

The preceding investigation of experimental alloys indicated that the nominal composition of the best alloy tested was as follows:

Magnesium	6 per cent
Manganese	1 per cent
Copper	1.5 per cent
Vanadium	0.1 per cent
Zirconium	0.05–0.25 per cent
Titanium	0.08 per cent
Beryllium	0.005 per cent
Aluminum (99.5 per cent)	balance

In order to compare the tensile and creep properties of this alloy with 142 and with the data available in the literature on Alcoa 254 alloy, a series of heats was prepared, heat treated, stabilized, and tested. The tensile properties were obtained at room temperature, 400, and 600 F., and creep properties were evaluated at 600 F. The compositions of the alloys are listed in Table V. Table VI shows the data on the heat treatments used, and Table VII shows the stabilization treatments applied to the alloys before testing at elevated temperature. The tensile properties at room temperature, 400, and 600 F. are listed in Table VIII, and the creep data obtained at 600 F. are shown in Table IX. Figure 6 shows the time-deformation curves for the experimental 6 per cent magnesium alloy, whereas Fig. 7 shows similar curves for Alcoa 142-T61 alloy.

TABLE VI.—HEAT TREATMENTS APPLIED TO THE ALLOYS LISTED IN TABLES VIII AND IX AND REPRESENTED BY FIGS. 6 TO 10.

Heat	Solution Treatment	Aging Treatment
	6 PER CENT MAGNESIUM ALLOY	
3991 3992 4208 4298	16 hr. at 810 F., quenched in cold water	None
	ALLOY NO. 142	
3993 3994 4299	8 hr. at 960 F., quenched in water at 150 to 212 F.	4 hr. at 400 F.

TABLE VII.—STABILIZATION TREATMENTS APPLIED TO ALLOYS LISTED IN TABLES VIII AND IX AND REPRESENTED BY FIGS. 6 TO 10.

Stabilization Treatment	Properties Determined
None	Tensile properties at room temperature
72 hr. at 400 F.	Tensile properties at room temperature and 400 F.
48 hr. at 500 F.	Tensile properties at room temperature and 400 F.
48 hr. at 600 F.	Tensile properties at room temperature and 600 F. and creep tests on 142 alloy at 600 F.
24 hr. at 650 F.	Tensile properties at room temperature and 600 F., creep at 600 F.
24 hr. at 700 F.	Tensile properties at room temperature and 600 F.

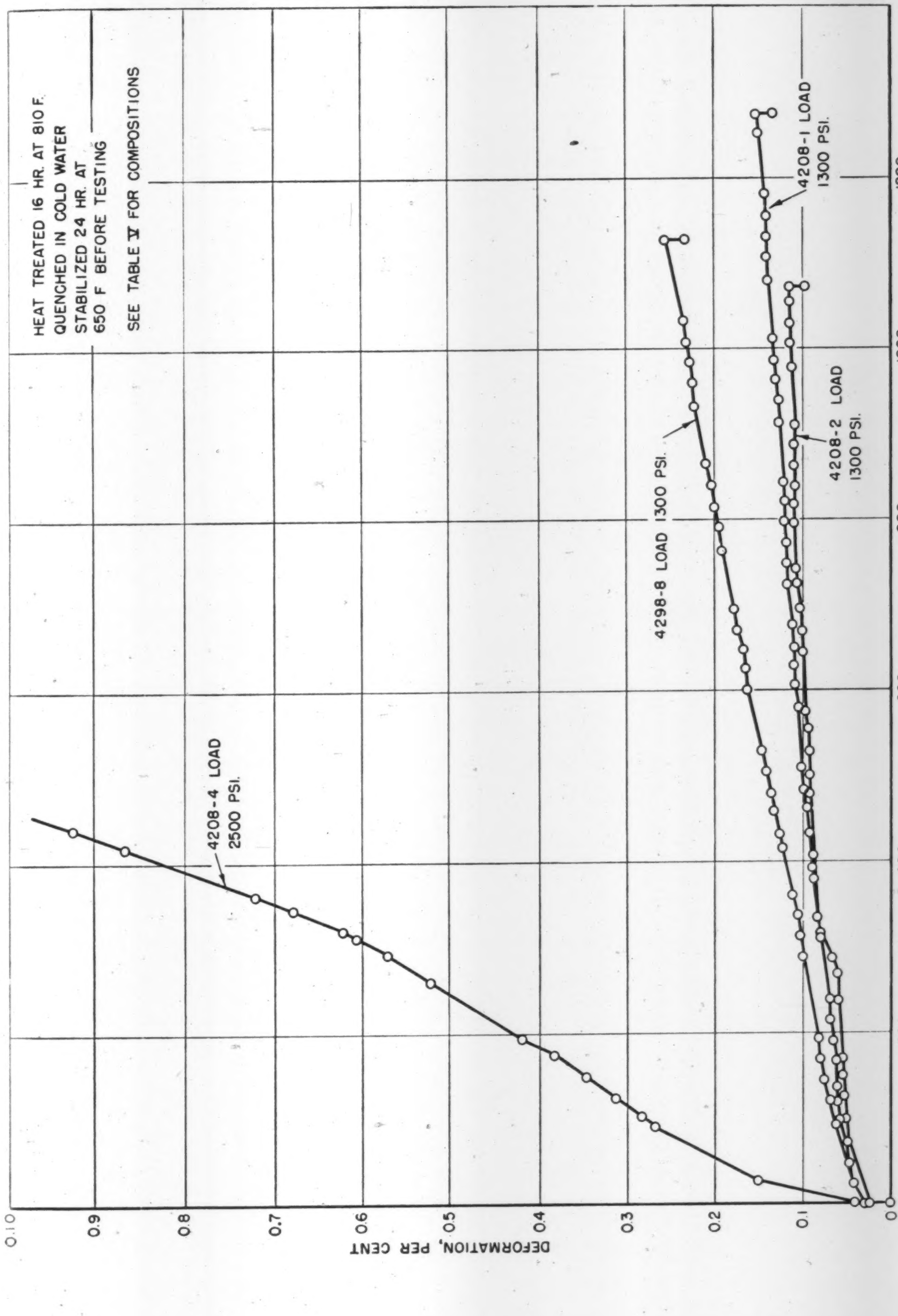

Fig. 6.—Time-Deformation Curves for 6 per cent Magnesium-Aluminum-Base Alloy at 600 F.

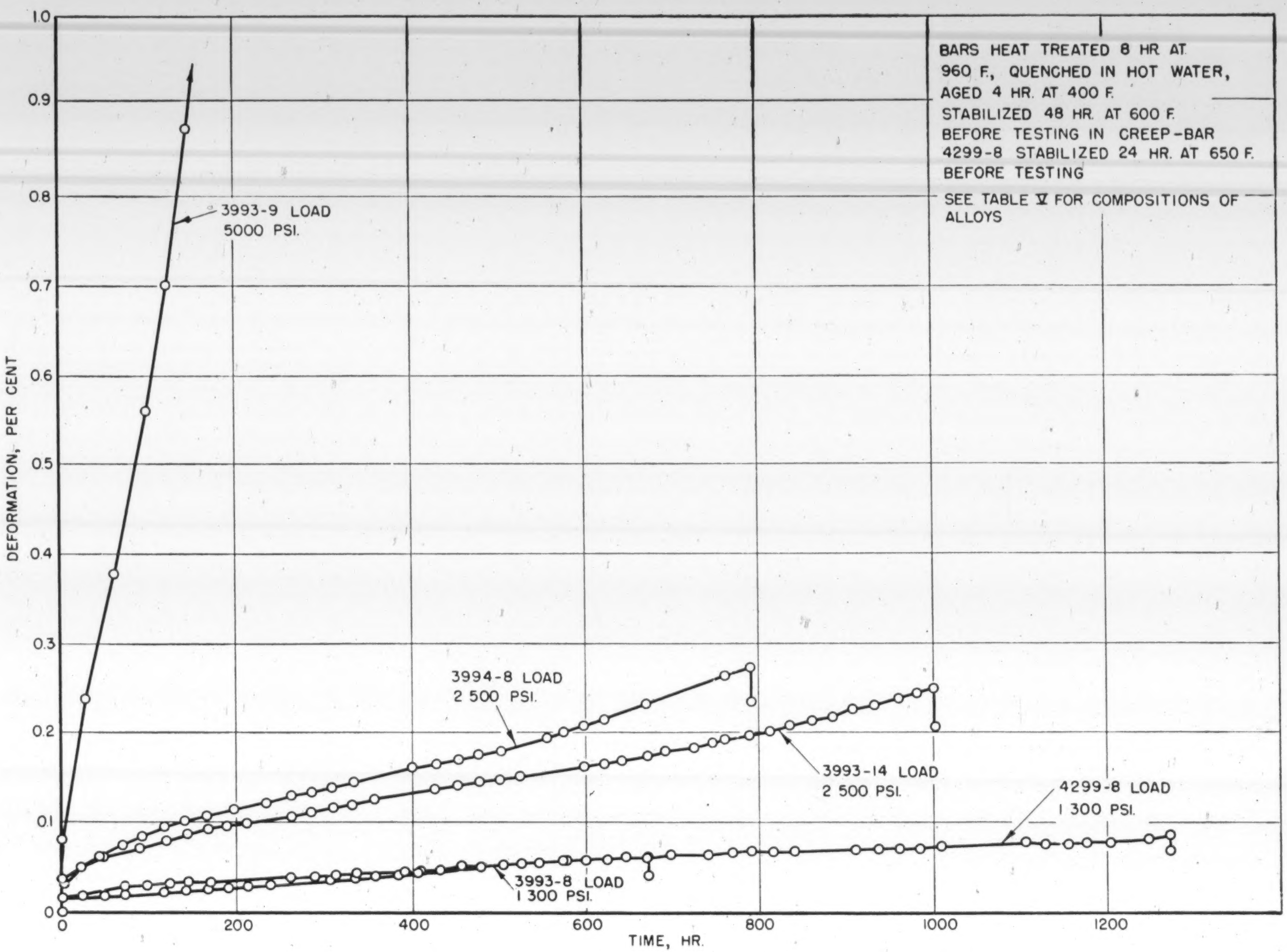

Fig. 7.—Time-Deformation Curves for Alloy 142-T61 Tested in Creep at 600 F.

TABLE VIII.—TENSILE PROPERTIES OF STABILIZED EXPERIMENTAL-6 PER CENT MAGNESIUM ALLOY AND 142 ALLOY AT ROOM AND ELEVATED TEMPERATURES.

Heat	Alloy	Heat Treatment	Aging	Stabilization Treatment	Tensile Properties[a] at Room Temperature				Tensile Properties[a] at 400 F.				Tensile Properties[a] at 600 F.			
					Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent	Tensile Strength, psi.	Yield Strength, psi.	Elongation in 2 in., per cent	Reduction of Area, per cent
4298	Experimental	16 hr. at 810 F.[b]	None	None	30 800	24 400	1.0	3.5	...	...	...	...	...	...	...	...
4299					44 400	43 100	1.0	1.0	...	...	...	...	...	...	...	...
3993	142	8 hr. at 960 F.[c]	4 hr. at 400 F.		35 000	34 100	1.0	...	...	...	...	...	...	...	...	...
3994					37 900	35 900	1.0	...	...	...	...	...	...	...	...	...
3991	Experimental	16 hr. at 810 F.[b]	None	72 hr. at 400 F.	39 400	30 500	2.0	...	29 000	24 200	5.0	8.0	...	...	...	...
3992					37 500	32 300	2.0	...	29 300	24 500	6.0	7.5	...	...	...	...
3993	142	8 hr. at 960 F.[c]	4 hr. at 400 F.		...	...	...	...	34 800	34 300	1.0	0.5	...	...	...	...
3994					...	...	...	...	34 700	...	1.0	...	...	...	...	...
3991	Experimental	16 hr. at 810 F.[b]	None	48 hr. at 500 F.	33 000	25 400	1.5	...	28 500	22 200	10.0	10.0	...	...	...	...
3992					30 700	27 000	1.5	...	27 100	21 600	3.0	3.5	...	...	...	...
3993	142	8 hr. at 960 F.[c]	4 hr. at 400 F.		36 800	...	1.0	1.0	31 400	29 400	1.5	1.0	...	...	...	...
3994					34 500	...	1.0	0.5	30 900	30 000	1.5	1.0	...	...	...	...
3991	Experimental	16 hr. at 810 F.[b]	None	48 hr. at 600 F.	38 500	24 700	3.5	...	...	...	...	...	13 900	9 900	75.0	58.5
3992					33 200	25 100	2.0	...	...	...	...	...	14 000	10 700	55.5	46.0
3993	142	8 hr. at 960 F.[c]	4 hr. at 400 F.		26 900	19 400	2.0	2.5	...	...	...	...	13 500	9 900	8.0	9.0
3994					24 500	16 900	2.0	2.5	...	...	...	...	13 200	9 200	10.5	9.5
4298	Experimental	16 hr. at 810 F.[b]	None	24 hr. at 650 F.	34 800	23 300	2.5	4.0	...	...	...	...	13 700	10 700	52.5	42.5
4299	142	8 hr. at 960 F.[c]	4 hr. at 400 F.		25 000	...	2.0	2.5	...	...	...	...	12 400	8 200	20.5	19.5
3991	Experimental	16 hr. at 810 F.[b]	None	24 hr. at 700 F.	32 400	24 300	2.0	3.0	...	...	...	...	15 300	10 900	35.0	37.5
3992					28 500	25 200	2.0	2.5	...	...	...	...	14 100	10 700	48.5	39.0
3993	142	8 hr. at 960 F.[c]	4 hr. at 400 F.		23 600	15 600	2.5	3.0	...	...	...	...	10 800	7 400	15.0	15.0
3994					24 600	16 700	3.5	4.5	...	...	...	...	10 400	6 300	22.5	21.5

[a] Average of two or three 0.505-in. machined tensile bars. Yield strength at 0.2 per cent offset.

[b] Quenched in cold water.

[c] Quenched in water at 150 to 212 F.

[d] See Table V for analysis of the alloys.

Tensile Properties.—Examination of the tensile data in Table VIII shows the following:

1. The room-temperature tensile properties of the heat-treated experimental aluminum-6 per cent magnesium alloy are improved by aging treatments of 72 hr. at 400 F. The tensile properties at room temperature are not damaged, or possibly are slightly improved by stabilization treatments of 24 hr. at 650 F., of 38 hr. at 500 F., or of 24 hr. at 700 F.

TABLE IX.—CREEP AND STRESS RUPTURE DATA ON EXPERIMENTAL-6 PER CENT MAGNESIUM ALUMINUM-BASE ALLOY AND 142 ALLOY AT 600 F.

Heat	Heat Treatment	Test Conditions		Minimum Creep Rate, per cent per hr.	Final Creep Rate, per cent per hr.	Transition to Third-Stage Creep		Elongation and Reduction of Area at Rupture		Time, hr., for Deformation of						Duration of Test, hr.
		Temperature, deg. Fahr.	Stress, psi.			Time, hr.	Deformation, per cent	Elongation, per cent	Reduction of Area, per cent	0.05 per cent	0.1 per cent	0.2 per cent	0.5 per cent	1.0 per cent	2.0 per cent	
EXPERIMENTAL ALLOY[d]																
4208	a	600	1300	0.00005	0.00005	...	...	...	...	75	675	...	...	...	...	1073
4208	a	600	1300	0.00007	0.00007	...	...	...	...	100	500	2000[d]				1277
4298	a	600	1300	0.00018	0.00018	...	...	...	...	55	300	825	2400[e]			1025
4208	a	600	2500	0.0015	>0.0055	295	0.58	6.5	14.5	3	15	55	248	455	738	845[f]
4208	a	600	5000	...	...	...	...	96	80	...	...	...	...	...	...	13.7[f]
ALLOY NO. 142-T61																
3993	c	600	1300	0.000072	0.000072	...	...	...	...	490	1200[e]					673
4299	b	600	1300	0.00003	0.00003	...	...	...	...	460	1600[e]					1270
3993	c	600	2500	0.00016	0.00019	...	...	...	...	20	200	810	2400[e]			1000
3994	c	600	2500	0.00022	0.00032	470	1.75			22	130	575				792
3993	c	600	5000	0.0048	>0.030	125	0.70	6.4	10.0	...	3	23	88	165	228	282[f]

[a] Heat treated 16 hr. at 810 F.; stabilized 24 hr. at 650 F. before testing in creep at 600 F.
[b] Heat treated 8 hr. at 960 F.; aged 4 hr. at 400 F.; stabilized 24 hr. at 650 F. before testing in creep at 600 F.
[c] Heat treated 8 hr. at 960 F.; aged 4 hr. at 400 F.; stabilized 48 hr. at 600 F. before testing in creep at 600 F.
[d] See Table V for analysis.
[e] Estimated values.
[f] Broke.

2. The room-temperature tensile properties of 142-T61 are very adversely affected by stabilization treatment of 48 hr. at 600 F., of 24 hr. at 650 F., or of 24 hr. at 700 F.

3. After stabilization at 600, 650, or 700 F., the room-temperature tensile properties of the aluminum-6 per cent magnesium alloy are superior to those of 142-T61 given a similar stabilization treatment.

4. The tensile properties of the experimental aluminum-6 per cent magnesium alloy at 400 F. after stabilization for 48 hr. at 500 F. averaged about as follows:

Ultimate strength, psi	28 000
Yield strength at 0.2 per cent offset, psi	22 000
Elongation in 2 in. per cent	6
Reduction in area, per cent	7

In contrast to this, Alcoa 142 alloy given the same stabilization treatment possessed the following properties:

Ultimate strength, psi	31 000
Yield strength at 0.2 per cent offset, psi	29 500
Elongation in 2 in. per cent	1.5
Reduction in area, per cent	1

According to published accounts,[3] the tensile properties of stabilized 142 alloy at 400 F. are as follows:

Ultimate strength, psi	22 000
Yield strength at 0.2 per cent offset, psi	16 000
Elongation in 2 in., per cent	2

Obviously, the Alcoa 142 alloy was not completely stabilized prior to the tension test at 400 F.

5. Tensile properties of both alloys at 600 F. are comparable when they have

been given the incomplete stabilization treatment of 48 hr. at 600 F. The experimental alloy, however, has a considerably higher elongation value.

6. When both alloys are given a more complete stabilization treatment of 24 hr. at 650 F., the experimental aluminum-6 per cent magnesium alloy has 1300 psi. greater ultimate strength and 2500 psi. greater yield strength, with higher elongation at 600 F. After the more complete stabilization treatment of 24 hr. at 700 F., the aluminum-6 per cent magnesium alloy has about 4000 psi. greater ultimate and yield strength and higher elongation than the 142 alloy at 600 F.

7. It is evident that the superiority of the tensile properties of the aluminum-6 per cent magnesium alloy becomes more pronounced as the stabilization of the alloy at the test temperature becomes more complete. This is to be expected because the limit of solid solubility of magnesium is near 6 per cent at 600 to 650 F.

Creep Properties.—The creep data in Table IX and the graphical representation of the creep data in Figs. 8, 9, 10, and 11 show the following:

1. The minimum creep rate obtained at a 1300 psi. load at 600 F. on both Alcoa 142 and the experimental aluminum-6 per cent magnesium alloy are approximately the same.

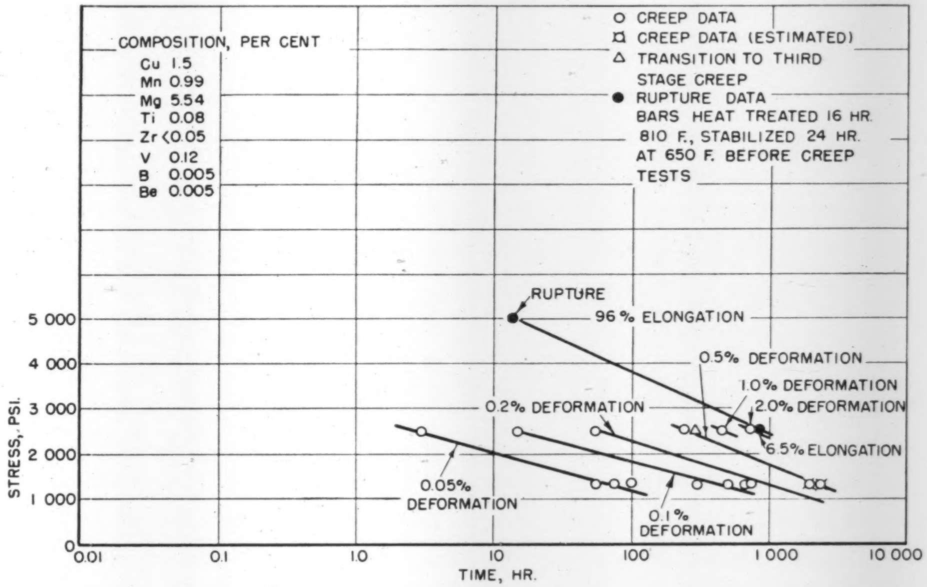

FIG. 8.—Stress *versus* Time for Total Deformation Curves for Experimental 6 per cent Mg-Al Base Alloy at 600 F.

2. At higher stresses, the Alcoa 142 is superior to the experimental aluminum-6 per cent magnesium alloy which, in turn, is superior to Alcoa 254.

3. As shown by Figs. 8, 9, and 10, for a total deformation of 0.5 per cent in 100 hr., the alloys at 600 F. will sustain the following loads:

Alcoa 254, psi.	2200
Experimental aluminum-6 per cent magnesium alloy, psi	3000
Alcoa 142, psi.	4900

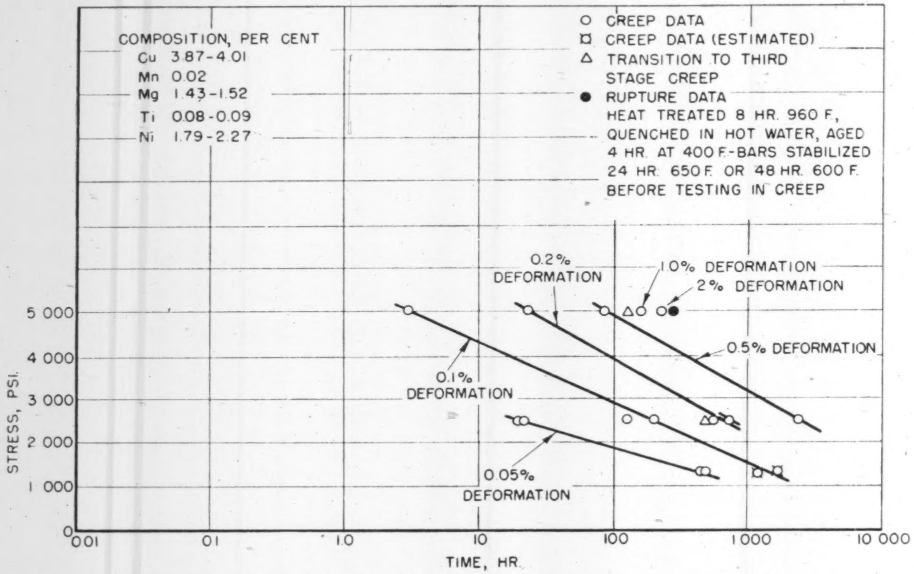

FIG. 9.—Stress *versus* Time for Total Deformation Curves for 141 – T61 at 600 F.

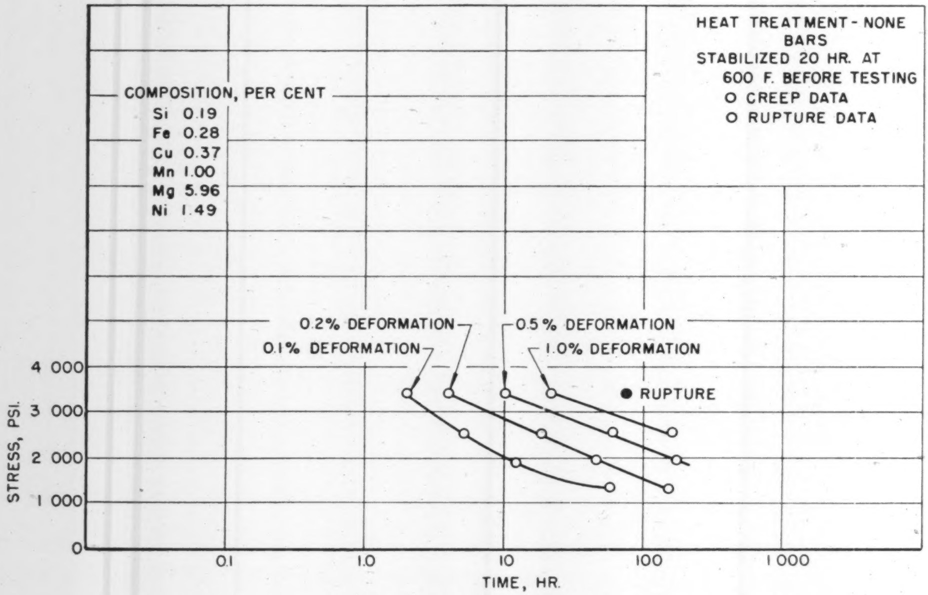

FIG. 10.—Stress *versus* Time for Total Deformation for Alloy 254 at 600 F.
See footnote 5 for reference.

SUMMARY

As a result of development work on casting alloys of aluminum containing 6 per cent magnesium, the following composition was developed for castings at elevated-temperature service:

Magnesium	6 per cent
Manganese	1 per cent
Copper	1.5 per cent
Vanadium	0.1 per cent
Zirconium	0.05 to 0.25 per cent
Titanium	0.08 per cent
Beryllium	0.005 per cent
Aluminum (99.5 per cent)	balance

This alloy has the advantages of not being seriously affected by stabilization at the temperature of test of 400 to 700 F. As a result, its tensile properties at 600 F. are high after stabilization at the test temperature. In addition, its density is low, its resistance to corrosion is excellent, and the machining characteristics are satisfactory. On the other hand, as compared with 142 alloy, the experimental alloy has somewhat poorer foundry characteristics. In addition, its thermal conductivity is substantially lower, which probably limits its use to applications where heat dissipation is unimportant. The creep properties of the experimental alloy are better than those of previously known aluminum-6 per cent magnesium alloys but somewhat inferior to 142 alloy. It is believed that additional improvement in the resistance to creep of the aluminum-6 per cent magnesium alloys could be obtained. If this were done, however, the load employed in the creep test should be raised fron 1300 psi. to 2500 psi. Under these conditions, improvements in resistance to creep produced by changes in the alloy composition can be more readily measured.

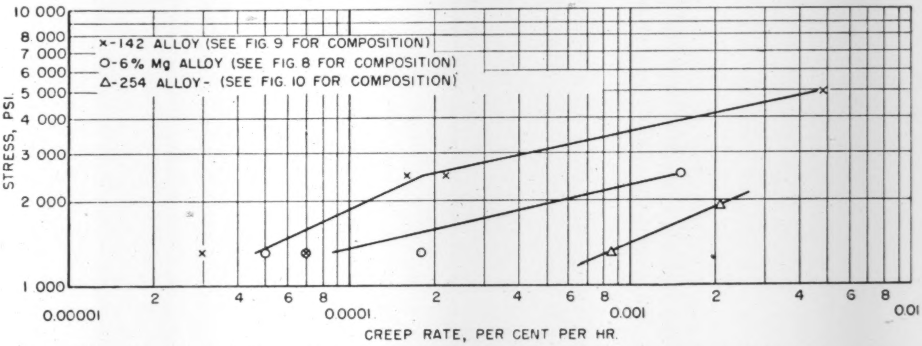

FIG. 11.—Stress *versus* Creep Rate Curves for Alloy 142 – T61 and 6 per cent Magnesium-Aluminum Base Alloys at 600 F.

See footnote 5 for reference.

Acknowledgment:

The authors gratefully acknowledge the support of the National Advisory Committee for Aeronautics who made this investigation possible and who granted permission for its publication, the assistance of H. C. Cross and Ward Simmons, who conducted the creep tests, and of Ray Kaiser and R. V. Whittenburg, who conducted much of the other laboratory work.

DISCUSSION

Mr. Walter Weil.[1]—I should like to know whether any figures are available on the fatigue strength of these aluminum-magnesium alloys at elevated temperature.

Mr. L. W. Eastwood (*author*).—In the work that I have reported, no data on fatigue were obtained. There are some data, however, on the effects of temperature on the properties of aluminum-base alloys. The best assemblage of such data is in a recent *Rand Report R-104* obtainable from the Rand Corp., Santa Monica, Calif.

There is, in addition, a recent paper in the British Institute of Metals which considers the fatigue properties of some of the alloys of interest in England.

Mr. J. T. Richards.[2]—Small beryllium additions are made to certain magnesium-bearing aluminum alloys to retard dross formation. Larger additions have been made on an experimental basis in attempts to increase the hardness and modulus of aluminum. In the investigation reported, 0.005 per cent beryllium was added to lessen the sand reaction in the mold.

Were any tests conducted to determine the optimum beryllium content or was the 0.005 per cent addition arbitrarily selected? Was any work done on the hardening effect of small beryllium additions?

Mr. Eastwood.—We used 0.005 per cent beryllium in the 6 per cent magnesium-aluminum base alloys in order to prevent sand reaction. We knew from previous experience that the 0.005 per cent beryllium was the correct amount. As far as I know, this amount of beryllium has no other effect upon the alloy.

We tried beryllium in the 6 per cent cerium-magnesium base alloy. The liquid solubility of beryllium in alloys of this type is very low. Consequently, it is difficult to add. We obtained varying results with the beryllium, particularly on creep resistance.

Higher amounts of beryllium in aluminum produces higher modulus of elasticity. However, it is a linear effect, and, in order to obtain appreciable increases in modulus, it is necessary to use large amounts of beryllium. At 25 per cent beryllium, the modulus of elasticity is 16 to 17 million psi.

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[1] Cleveland, Ohio.

[2] Development Engineer, The Beryllium Corp., Reading, Pa.

TENSILE PROPERTIES OF SOME AIRCRAFT STRUCTURAL MATERIALS AT VARIOUS RATES OF LOADING*

BY RICHARD F. KLINGER[1]

SYNOPSIS

The effect of rate of loading in tension was determined at room temperature on non-metallic and metallic aircraft structural materials. Stress-strain curves and ultimate strength values were obtained over the range of times to reach yield or ultimate strengths of 0.003 sec. to 2 hr., and rate of loading-strength relationships are presented. The results indicate a pronounced increase of ultimate and yield strength values with increased rate of loading for plastic materials such as polymethylmethacrylate cast sheet, cotton fabric base phenolic laminate, and glass fabric base polyester laminate. There was also a marked increase in initial modulus of elasticity with increase in rate of loading for the polymethylmethacrylate material. The test results for the metals, bare 24S-T and bare 75S-T aluminum alloys showed little change in yield strengths with respect to rate of loading.

Many materials used in aircraft structures are subject to high rates of loading developed by high-speed maneuvers and sudden atmospheric changes (gusts) which rapidly produce high stresses of short duration. These loading times have been reported to be as short as 0.01 sec. It is, therefore, desirable to have an understanding of the relationship between rates of loading as encountered in aircraft and the stress-strain characteristics of the material. Therefore tension tests were conducted on three non-metallic and two metallic materials used in aircraft to determine the relationship of loading time to strength.

MATERIALS

The principal structural plastic materials used in aircraft are (1) low-pressure glass fabric base polyester laminates for radio and radar antenna housings, wing tips, ducts, etc., (2) polymethylmethacrylate for canopies and other glazing purposes, and (3) postformed miscellaneous interior parts made from high-pressure cotton fabric base phenolic laminate post forming stock. Material representative of each of these was included in the project.

The material consisted of the following sheets: $\frac{1}{8}$ in. low-pressure-laminated glass fabric-polyester resin, $\frac{1}{8}$ in. high-pressure-laminated cotton fabric phenol-formaldehyde resin, $\frac{1}{4}$ in. cast methylmethacrylate and bare 0.064 in. 24S-T3 and 75S-T6 aluminum alloys.

The polymethyl methacrylate was transparent material conforming to Specification AN-P-44. The glass fabric base plastic laminate consisted of 13 plies of 181-114 satin weave fabric

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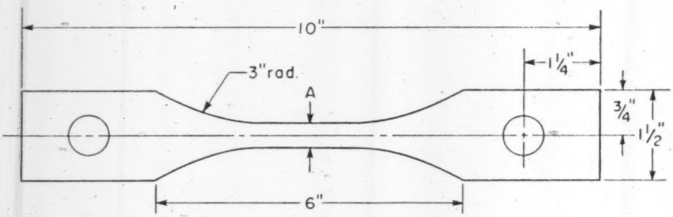
* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950

[1] Wright-Patterson Air Force Base, Dayton, Ohio.

parallel laminated with a low-viscosity alkyd-styrene type polyester, Selectron 5003. The resin was catalyzed by 1.6 per cent by weight of Luperco ATC mixture of tricresyl phosphate and benzoyl peroxide (each 50 per cent by weight of the mixture) contining 3.30 per cent active oxygen. The lay-up was made between cellophane covered glass plates. Immediately after impregnation and lay-up, the panel was curved at a pressure of 13 psi. for $1\frac{1}{2}$ hr. in an oven at 265 F. Pressure was applied by an air-inflated rubber diaphragm located on the bottom plate of the press. The average resin content and specific gravity of the panel were 35 per cent and 1.89, respectively.



	A
Methylmethacrylate	0.250 in.
Glass fabric laminate	0.250 in.
Cotton fabric laminate	0.500 in.

FIG. 1.—Tension Specimen Used For Rapid Loading Tests of Plastics. Holes $\frac{33}{64}$ in. in Diameter.

The cotton fabric base phenolic plastic laminate was a typical aircraft post-forming stock, Formica Grade CJP-11. The fabric base had the following specifications: weight 8.15 oz. per sq. yd.; moisture content, $6\frac{1}{2}$ per cent; crimp, warp—12 per cent, filling—9 per cent; yarn, warp—single yarn, filling—single yarn; count, warp—41 $\pm \frac{1}{2}$ per cent, filling—37 per cent; staple, strict middling grade—$\frac{15}{16}$ to 1 in. in length; tensile strength, 110 lb. per in. with 15 per cent elongation at break, filling—105 lb. per in. with 15 per cent elongation at break tested by grab method. The resin was a modified phenolic bearing the Bakelite Manufacturing Co.'s No. 17090. The material was cured at a temperature of 155 C. and 110 psi. pressure. The volatile content before pressing was between 4 and 5 per cent based on a drying temperature of 150 F. for approximately 12 min. The resin content of the finished laminate was approximately 50 per cent.

PROCEDURE

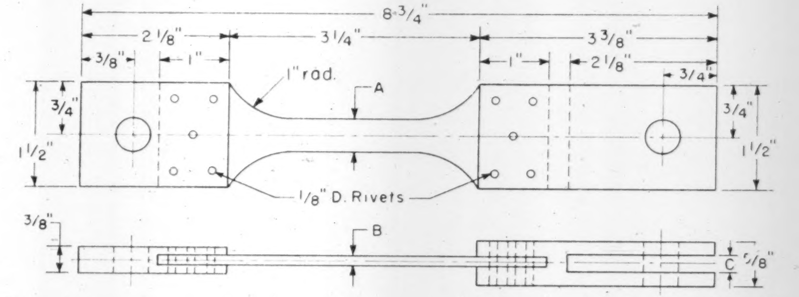
Plastic Materials:

Specimens of the material were as shown in Fig. 1. All specimens were cut and machined from the sheet in the direction of greater tensile strength. The width of the gage section of specimens of glass fabric laminate and methylmethacrylate sheets was 0.250 in. and that of the cotton fabric laminate sheet was 0.500 in.

Tests were conducted in a Baldwin-Southwark universal testing machine and Tinius Olsen combination impact testing machine. By using the head motor in conjunction with the hydraulic loading system of the universal testing machine, tests of 0.3 sec. in duration, through yield strength, and longer were possible. The impact testing machine with special jig for tensile loading and rubber shock absorbers produced tests in the vicinity of 0.003 sec. in duration.

Strains at normal speeds of loading were measured with a 2-in. gage length Tinius Olsen last word extensometer (0.0001 in. division). At higher loading speeds (0.003 to 300 sec. to yield strength) the Baldwin Southwark SR-4 type A-17-2 electrical-resistance strain gage was used to indicate strain. This gage has a length of 1 in., a resistance of

1020 ohms, a strain sensitivity of 2 and a measuring range of from 0 to 0.015 in. per in. As a load-indicating device the dynamometer shown in Fig. 2 was placed in series with the specimen. The dynamometer consisted of 24S-T aluminum alloy sheet machined to the form of a tension specimen which was riveted at each end to aluminum alloy connecting blocks. As indicated in the figure, dynamometers of different cross-sections were used for each material tested to develop maximum strains without exceeding the elastic limit of the 24S-T


8 3/4"
2 1/8"
3 1/4"
3 3/8"
3/8"
1"
1"
2 1/8"
3/4"
1" rad.
A
3/4"
1 1/2"
3/4"
1 1/2"
1/8" D. Rivets
3/8"
B
C
5/8"

For testing:	A	B	C
Methylmethacrylate	0.375 in.	0.064 in.	5/16 in.
Glass fabric laminate	0.520 in.	0.125 in.	5/32 in.
Cotton fabric laminate	0.700 in.	0.032 in.	1/8 in.
Aluminum alloys (24S-T, 75S-T)	1.00 in.	0.067 in.	1/16 in.

FIG. 2.—Dynamometer Used For Rapid Loading Tests.
2 Holes 33/64 in. in Diameter.

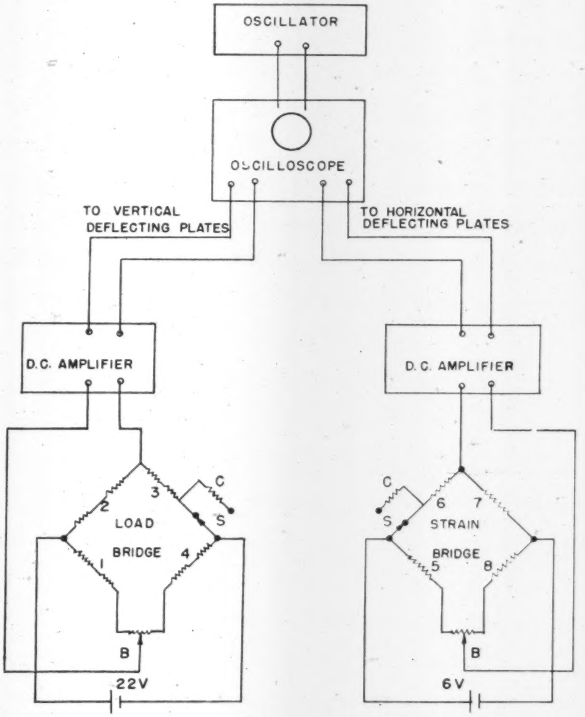

OSCILLATOR
OSCILLOSCOPE
TO VERTICAL DEFLECTING PLATES
TO HORIZONTAL DEFLECTING PLATES
D.C. AMPLIFIER
D.C. AMPLIFIER
LOAD BRIDGE
1
2
3
4
C
S
B
22V
STRAIN BRIDGE
5
6
7
8
C
S
B
6V

FIG. 3.—Rapid Loading Electrical Recording System.

alloy when maximum specimen loads were applied.

A diagram of the basic electrical recording circuits employed is given in Fig. 3. Each arm of the load-indicating bridge consists of two A-17-2 gages in series. The gages of arm 1 were glued side by side to one face of the dynamometer in the center of the gage section and the gages of arm 3 were glued similarly to the opposite face. The remaining gages in arms 2 and 4 were glued to a piece of $\frac{1}{16}$-in. sheet aluminum alloy for stability. The strain-indicating bridge contained one gage per arm, and the gages were applied in the same manner as those in the load-indicating bridge to the test specimens instead of the dynamometer. No precautions were taken to compensate for temperature changes during tests because test durations were short.

All gluing was accomplished with "Duco" cement (cellulose nitrate plus a solvent) in the manner recommended by the strain gage manufacturer and as recommended by Rosato (**2**)[2] for methylmethacrylate sheet. In work at the Naval Air Material Center, Rosato found that careful application of the gages caused insignificant effects on the tensile properties of acrylate sheet. This method involved spreading the cement only on the gage, then applying the gage to the specimen, and drying at room temperature. Any specimens showing the slightest sign of crazing were discarded, even though a small amount of crazing has negligible effect on tensile yield and ultimate strengths of acrylate sheet. In addition to the gages on the specimens, each bridge contained a balancing resistance, *B*, calibrating resistance, *C*,

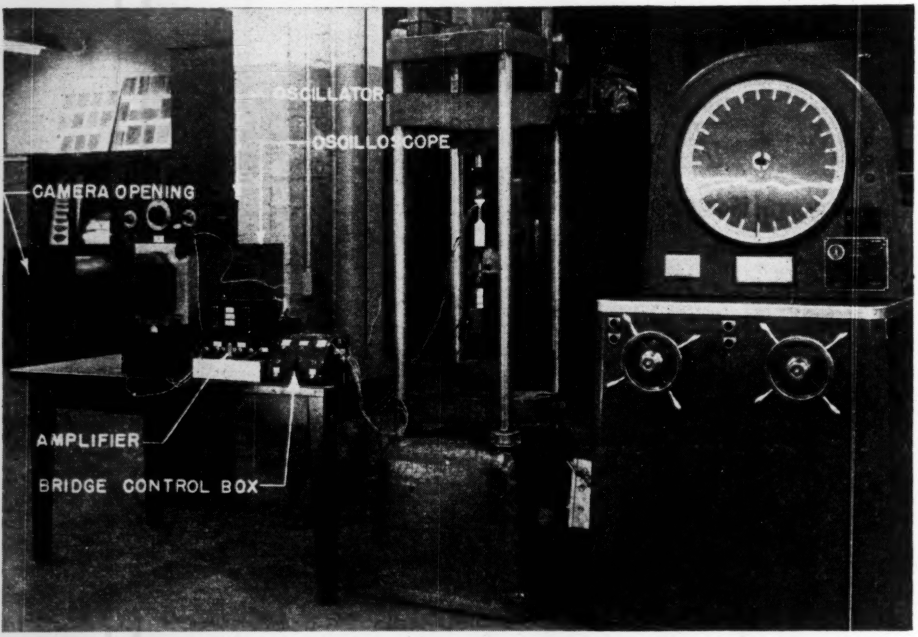


FIG. 4.—Apparatus for Rapid Loading Tension Tests.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1050.

and switch, *S*, for introducing the calibrating resistance. The bridges were powered by separate batteries and the unbalance of each bridge was passed through a direct current amplifier and then to an RCA Type 327-A cathode-ray oscilloscope.

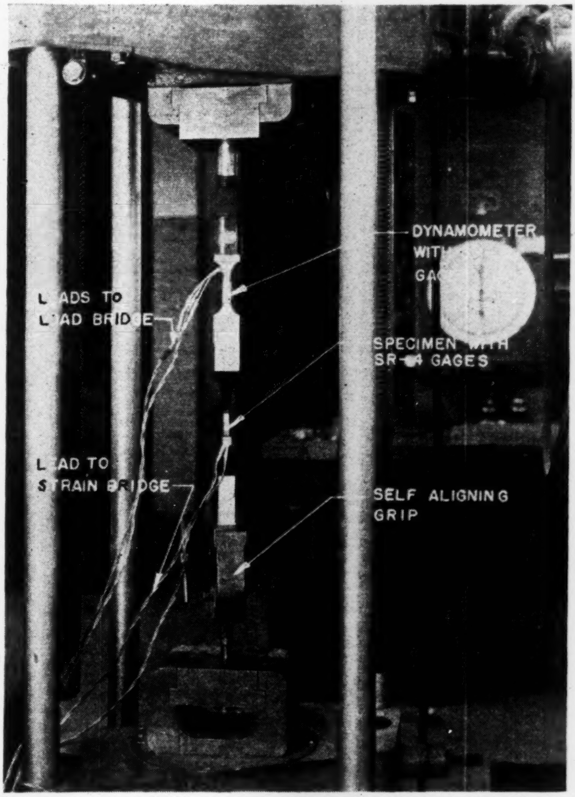


FIG. 5.—Specimen and Dynamometer Set-up in Universal Testing Machine.

The signal from the load-indicating bridge was impressed on the vertical deflecting plates of the oscilloscope and that from the strain indicating bridge on the horizontal deflecting plates. When load is applied to the specimen and dynamometer, the signals from the two bridges are added to provide a trace of the regular load-strain curve on the oscilloscope tube. The oscilloscope has a 9-in. diameter cathode-ray tube and provides a convenient method for measuring d-c. voltages. To eliminate most of the distortion caused by the curvature of the face of the tube, only the center section of the tube was utilized. This provided a relatively flat surface in the form of a square with 4-in. sides. As a means of determining the test duration a Hewlett Packard Audio Oscillator Model 200B was inserted in the circuit of the oscilloscope which has provisions for modulation of its electron stream.

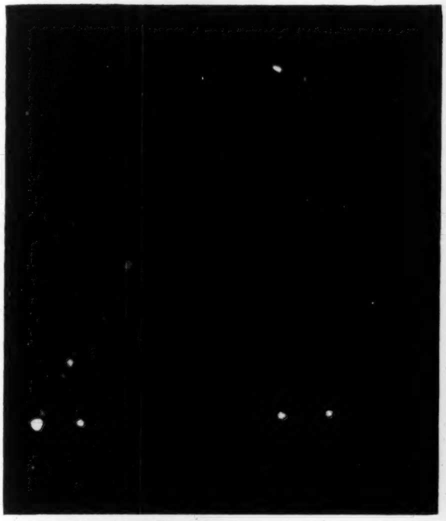
The traces provided by the oscilloscope were photographed using an 8 by 10 in. view camera with an f2.5 lens and Kodak Super XX and Tri X Panchromatic film. Photographs were taken in the dark by enclosing the space between camera lens and oscilloscope screen in a cardboard tube with blackened inner surfaces. Figure 4 gives a general view of the testing layout and Fig. 5, a view of specimen and dynamometer in the universal testing machine.

The calibration of the strain-indicating bridge was accomplished in the following manner: A specimen, with SR-4 gages attached and incorporated in the proper bridge, was placed in the universal testing machine using self-aligning grips without the dynamometer. A Tinius Olsen Last Word extensometer with 1-in. gage length and 0.0001-in. divisions was mounted on the edge of the specimen straddling the SR-4 strain gages. With the bridge balanced and equipment ready for operation, the calibrating resistance was switched into its arm of the bridge providing an unbalance and causing the dot on the screen to trace a horizontal path across the face of the tube which was photographed. The dot was then moved approximately $\frac{1}{2}$ in. in the vertical direction and, with the calibrating resistance out of the circuit, load applied to the specimen until a preassigned strain was indicated by the extensometer. The trace thus produced was also photographed and the same procedure applied four times across the face of the tube. From the print of the film, the ratios of the traces were converted into an average strain value for the calibrating resistance. An average value of strain was used because the curvature of the tube produces a variation of approximately 2 per cent in displacement for a given signal as the face of the tube is traversed. The calibration procedure was applied to several specimens of each material before assigning a value to the calibrating resistance.

The calibration of the resistance in the load-indicating bridge was conducted in a similar manner except a fixed load, in the vicinity of the yield strength of the material for which the dynamometer was designed, was applied to the dynamometer in place of a given strain to the specimen. A series of displacement lines were photographed as for the strain calibration, and from the ratios of the resistance and dynamometer load displacements a load value was calculated for the calibrating resistance.

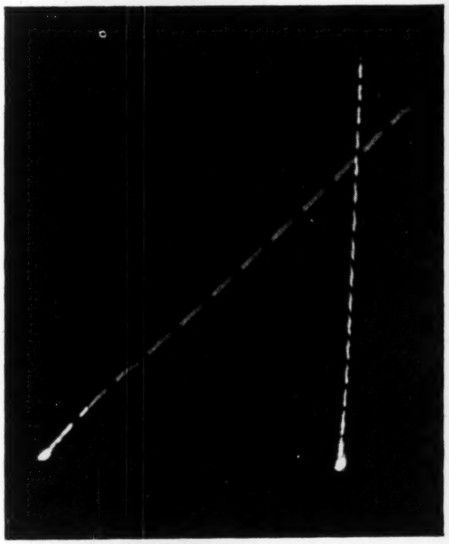
The sequence of operations for a test using electrical strain gages and oscilloscope is as follows: The specimen and dynamometer are connected in series by a $\frac{1}{2}$-in. bolt and the assembly inserted in one of the two testing machines by means of yokes and $\frac{1}{2}$ in. bolts. With the camera shutter open, switch S of the load bridge (Fig. 3) is closed, the shutter closed and then the switch opened. This action produces the vertical or load calibration line and the same procedure using the strain bridge provides the horizontal or strain calibration line.

A rate of loading is determined and the oscillator set at a frequency to give an appropriate number of interruptions in the diagonal trace. The dot is then located at the proper corner of the oscilloscope screen, and, with both calibrating switches and camera shutter open, the load is applied at the predetermined rate and, after failure of the specimen, the shutter closed. The yield strength is determined from the print of each test being taken at 0.01 in. per in. total strain for specimens of cotton fabric laminate and glass fabric laminate and 0.015 in. per in. total strain for methylmethacrylate specimens. Because the initial straight-line portion of the stress-strain curves of the plastic materials tested formed such a relatively small portion of the entire curve to the yield strength, it was difficult to definitely and accurately locate the initial line on



Specimen No. 27
Time to yield strength—0.63 sec.
Yield strength—1080 psi.
Frequency—30 cps.
Calibration—515 lb., 0.002 in. per in.
Gage section—0.122 by 0.402 in.

FIG. 6.—Load-Strain Curve of Cotton Fabric Laminate.



Specimen No. 28 (Left) Time to yield strength 0.30 sec. Yield strength—35,600 psi. Gage section—0.129 by 0.256 in.

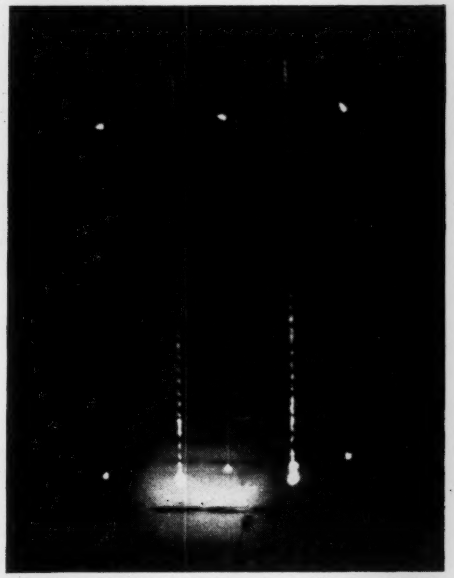
Specimen No. 27 (right). Time to tensile strength 0.53 sec.

Ultimate strength—68,500 psi. Gage section—0.120 by 0.254 in.

Frequency—30 cps. Calibration 1200 lb., 0.0068 in. per in.

FIG. 7.—Load-Strain Curve and Tensile Strength Test of Glass Fabric Laminate.

the individual relatively small oscilloscope records even though the slopes (moduli of elasticity) could be determined with fair accuracy. This rendered the determination of offset yield strength subject to excessive scatter. Therefore, a total strain was used to define the yield strength. However the yield strength of the cotton fabric laminate was also calculated at 0.2 per cent offset from the modulus line because considerably less scatter was obtained for offset yield strength of this material.



Specimen No. 19 (left)—Time to tensile strength 0.98 sec.

Ultimate strength—12,600 psi. Gage section —0.120 by 0.401 in.

Specimen 20 (right)—Time to ultimate strength 0.88 sec.

Ultimate strength—12,200 psi. Gage section 0.125 by 0.402 in.

Frequency 40 cps. Calibration 515 lb.

FIG. 8.—Tensile Strength Test of Cotton Fabric Laminate.

Since the strain range of the electrical strain gages is not great enough to allow the recording of the ultimate strength, additional specimens were required for these tests at the higher loading speeds. Ultimate strength specimens, free of gages, were placed in series with the dynamometer and tested in the same

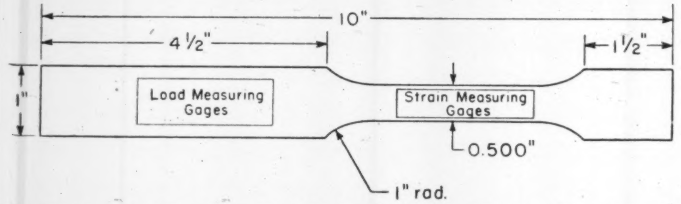


FIG. 9.—Tension Test Specimen Used for Rapid Loading Tests of Aluminum Alloys.

TABLE I.—YIELD AND ULTIMATE STRENGTHS OF 24S-T3 ALUMINUM ALLOY SHEET[a] AS DETERMINED SIMULTANEOUSLY FROM SAME SPECIMEN WITH OLSEN AND A17-2 SR-4 STRAIN GAGES WITH STATIC LOADING.

Specimen	Olsen Strain Gage		Oscilloscope Record			
			A17-2 SR-4 Strain Gage			
			Machine Calibration		Beam Calibration	
	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.	Yield Strength, 0.2 per cent Offset, psi.	Tensile Strength, psi.
No. 0L	52 900	62 600	b	b	52 900	60 200
No. 1L	54 600	59 000	b	b	52 200	56 300
No. 2L	53 800	71 800	b	b	52 500	69 000
No. 3L	54 000	68 000	52 500	70 900	52 400	69 700
No. 4L	...	...	c	c	c	c
No. 5T	46 200	66 700	48 300	68 600	47 600	67 400
No. 6T	46 000	70 400	47 800	71 100	47 600	70 300
No. 7T	...	...	c	c	c	c
No. 8T	46 400	70 300	49 500	67 000	47 300	68 400
No. 9T	46 000	69 000	46 100	68 000	46 200	67 000
No. 10T	45 700	69 500	45 000	69 400	43 600	67 200
No. 11T	...	...	c	c	c	c
No. 12T	43 500	69 200	45 300	67 300	44 500	66 300
No. 13L	55 000	71 800	52 800	c	53 900	c
No. 14T	46 200	69 500	46 100	68 600	45 800	68 000

T—Specimen taken in transverse direction.
L—Specimen taken in longitudinal direction.
[a] 0.064 in. bare sheet.
[b] No data collected.
[c] Photograph distorted.

manner as other specimens. The trace resulting is a straight vertical line proportional to the load applied to the dynamometer. The duration is taken as the reciprocal of the oscillator frequency times the number of dots to the yield or ultimate strength. Figures 6 through 8 present typical traces obtained at various loading speeds.

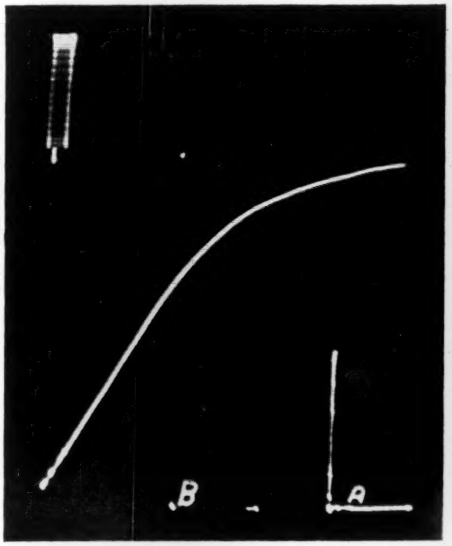
Aluminum Alloys:

Specimens of 75 S - T and 24 S - T aluminum alloys as shown in Fig. 9 were tested in the universal testing machine to determine the accuracy of the electrical gage—oscilloscope set-up. The lengthened grip section of the specimen acted as a dynamometer with gages glued to each side of the specimen as indicated in the figure. For the more rapid loading rates in the universal testing machine and impact machine, specimens as given in Fig. 9 but modified to provide for a $\frac{1}{2}$-in. diameter hole at each end were connected in series with the dynamometer shown in Fig. 2.

The test procedures for the metals were similar to those employed in testing the plastic materials with the exception of the method of calibration. A cantilever beam served as a means of calibration replacing the calibrating resistance of each bridge. The beam was made of 75 S - T aluminum alloy 12 by 1 by $\frac{5}{16}$ in. and was provided with a hand lever at its free end to give a set deflection when desired. Gages of arms 6 and 8 (Fig. 3) were mounted side by side 9 in. from the free end of the beam for strain calibration and gages of arms 1 and 3, 7 in. from the free end for load calibration.

Tests were conducted to compare results as obtained with electric strain gages and Olsen Last Word extensometer. A 1-in. gage length Olsen gage (0.0001 in. per in. per division) was mounted on the edge of the specimen (Fig. 12) straddling the A - 17 - 2 gages and readings were taken simultaneously with the recording of the electric strain gage data. Results of these tests are given in Table I. Basing the calculations on the Olsen gage data, the yield and ultimate strengths as determined from the A-17-2 gages and oscilloscope records were in error approximately 2.5 per cent.

Tests of the aluminum alloys at greater rates of loading were conducted in a Baldwin Southwark universal testing machine and a Tinus Olsen Change-O-Matic impact testing machine. Type C-1 SR-4 strain gages were used in place of the A-17-2 type in these tests, but the electrical system remained basically the same. Typical records of tests at different loading rates are shown in Figs. 10 and 11. The yield strengths were calculated with a 0.20 per cent offset.



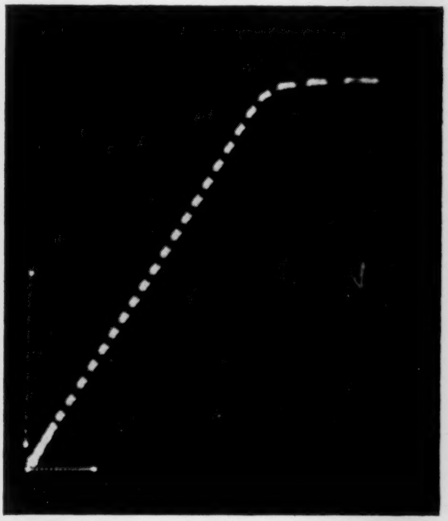
Test duration—about 5 min.
A—Displacements made by preloading specimen
B—Displacements made by bending calibrating beam

Olsen Gage Test Results	Yield Strength, psi.	Tensile Strength, psi.
Results using Beam Calibration...	46 200	67 000
Results using Preload Calibration.	46 100	68 000

FIG. 10.—Calibration Test of 24S-T Aluminum Alloy Sheet Specimen.

DISCUSSION

The results of all tests of non-metallic materials are provided in Tables II, III, and IV. The yield strengths were calculated on the basis of a total strain which is approximately equal to the average total strain at the yield strength as obtained by the 0.2 per cent offset method with normal static loading. The use of total strain to define the yield strength eliminated scatter arising from locating the short initial straight portion of the load-strain curve as a basis for the usual offset calculation as explained above. The relationship between the two types of yield calculations is shown in the data given for the cotton fabric laminate which was found more suitable for offset yield calculations, because of less scatter being obtained using the offset procedure than for the other materials. The average yield strength as calculated with a 0.2 per cent offset increased 80 per cent over the time to yield strength range of 2 hr. to 0.005 sec. and but 45



Time to yield strength—0.9 sec.
Yield strength, 72,200 psi.
Frequency of dashes—30 per sec.
Horizontal line represents 0.00204 in. per in. strain
Vertical line represents 570 lb.
Specimen gage section 0.066 by 0.500 in.

FIG. 11.—Rapid Loading Properties of 75S-T Aluminum Alloy Sheet.

per cent when calculated using a total strain of 0.01 in. per in.

The considerably smaller increase in yield strength obtained in the latter method of calculation is demonstrated in Fig. 12. Curve *A* in the figure represents a static test and (*a*) the offset yield strength of a given material. Curve *B* is the stress-strain curve of the same material but at a higher rate of loading. If a total strain equal to that present at the offset yield strength of the static test is used to define the yield strength, a lower value (*b*) of stress is obtained relative to the value (*c*) which would be the yield strength as calculated by the offset method. This example assumed a constant modulus while the plastic materials tested, in general, showed an increase in modulus with higher loading rates. However, the increase in modulus was not great enough to overcome the lowering of the yield strength obtained by the total strain method of calculation, and, therefore, a smaller increase in yield strength with increasing loading rates results.

TABLE II.—TENSILE PROPERTIES OF COTTON FABRIC—PHENOL FORMALDEHYDE LAMINATE AT VARIOUS RATES OF LOADING (⅛-IN. SHEET).

Specimen	Time to Tensile Strength, sec.	Tensile Setrength, psi.	Time to Yield Strength, sec.	Yield Strength, psi.	
				0.2 per cent Offset	0.01 in. per in. Total Strain
No. 1	9600	11 070	8100	6 850	7 800
No. 2	7900	11 030	4800	7 580	8 700
No. 3	380	11 600	300	6 880	7 600
No. 4	340	11 230	270	7 470	7 700
No. 5	310	10 820	270	7 380	8 000
No. 6	340	11 480	240	7 750	8 300
No. 7	310	10 880	270	6 690	7 830
No. 8	210	11 200			
No. 9	240	11 200			
No. 10	210	11 300	180	7 780	8 440
No. 11	240	11 000	180	8 140	8 680
No. 12	240	11 100	180	7 360	7 640
No. 13	5.3	11 900			
No. 14	7.0	11 700			
No. 15			6.1	8 670	8 770
No. 16	6.4	11 000			
No. 17			5.4	9 630	9 730
No. 18	6.7	11 700			
No. 19	0.98	12 600			
No. 20	0.88	12 200			
No. 21			1.3	11 000	10 600
No. 22	1.0	12 300			
No. 23			0.83	10 600	10 200
No. 24	0.93	12 000			
No. 25			0.66	11 100	10 300
No. 26	0.80	12 200			
No. 27			0.63	11 500	10 800
No. 28	0.73	12 100			
No. 29	0.68	12 100			
No. 30	0.70	12 700			
No. 31			0.36	9 500	8 750
No. 32			0.28	11 700	10 700
No. 33	0.42	12 600			
No. 34	0.39	13 100			
No. 35	0.42	12 200			
No. 36	0.0050	14 400			
No. 37	0.0040	14 800			
No. 38	0.0027	14 100			
No. 39			0.0065	12 800	11 700
No. 40			0.0030	11 800	11 100
No. 41			0.0056	14 700	12 200
No. 42			0.0062	13 200	12 100

TABLE III.—TENSILE PROPERTIES OF METHYL-METHACRYLATE AT VARIOUS RATES OF LOADING (¼-IN. SHEET).

Specimen	Time to Tensile Strength, sec.	Tensile Strength, psi.	Time to Yield Strength, sec.	Yield Strength at 0.015 in. per in. Strain, psi.
No. 1	8500	6 350	6600	4 380
No. 2	7200	6 630	4200	4 550
No. 3	430	8 300	330	5 570
No. 4	360	7 920	270	5 980
No. 5	390	7 450	300	6 210
No. 6	360	7 190	240	6 220
No. 7	30	8 210	20	6 020
No. 8	32	8 330	18	6 220
No. 9			1.9	6 420
No. 10	3	9 980		
No. 11			2.0	6 360
No. 12	2.9	9 970		
No. 13			1.0	6 650
No. 14	1.0	10 150		
No. 15			0.25	7 230
No. 16	0.46	9 100		
No. 17			0.32	7 260
No. 18	0.43	10 500		
No. 19			0.32	6 930
No. 20	0.45	10 100		
No. 21			0.005	9 200
No. 22			0.008	9 600
No. 23	0.0075	13 100		
No. 24			0.005	10 000
No. 25	0.006	13 200		
No. 26			0.0055	9 700
No. 27	0.004	13 500		
No. 28			0.007	10 000

The relationship between time and strength of the plastic materials is shown in Figs. 13, 14, and 15.

The yield and ultimate strengths of the cotton fabric laminate increase with loading speed, and when plotted against the logarithm of time approaches a straight-line relationship in the time range of from 4 min. to 0.005 sec. Slower loading speeds of 2 hr. produced no change in either yield or ultimate strength with respect to values obtained in 4-min. loading times. The increase in the ultimate strength over the complete time range was 30 per cent and 45 per cent for the yield strength.

TABLE IV.—TENSILE PROPERTIES OF GLASS FABRIC POLYESTER LAMINATE AT VARIOUS RATES OF LOADING (⅛-IN. SHEET).

Specimen	Time to Tensile Strength, sec.	Tensile Strength, psi.	Time to Yield Strength, sec.	Yield Strength at 0.01 in. per in. Strain, psi.
No. 1	10 000	45 600	8000	30 000
No. 2	8 900	40 700	7600	29 600
No. 3	390	55 300	330	30 800
No. 4	360	54 800	300	29 900
No. 5	330	53 500	300	30 600
No. 6	330	52 800	270	30 100
No. 7	300	53 900	240	28 600
No. 8	240	56 000	180	31 200
No. 9	240	56 500	180	36 300
No. 10	210	53 300	180	32 700
No. 11	240	52 400	210	36 800
No. 12	240	49 900	210	33 400
No. 13	210	56 000		
No. 14	27	53 000		
No. 15	30	56 400	18	32 900
No. 16	25	55 700		
No. 17			20	34 000
No. 18			3.2	35 100
No. 19	3.6	58 500		
No. 20			2.7	35 100
No. 21	3.3	58 000		
No. 22	3.6	56 600		
No. 23	0.80	60 800		
No. 24			0.66	36 000
No. 25			0.66	34 400
No. 26	1.2	64 300		
No. 27	0.53	68 500		
No. 28			0.30	35 600
No. 29			0.30	35 400
No. 30	0.47	72 800		
No. 31	0.53	64 400		
No. 32			0.0024	33 500
No. 33			0.0030	33 800
No. 34	0.004	63 500		
No. 35	0.005	62 900		
No. 36	0.004	57 700		
No. 37			0.0028	33 100
No. 38	0.0052	59 000		
No. 39	0.0052	60 200		
No. 40			0.0030	36 900
No. 41			0.0026	37 600

The yield strength of the methylmethacrylate increased 95 per cent and its ultimate strength 100 per cent over the complete time range. The increase in ultimate strength was approximately linear with the logarithm of time but the yield strength curve was divided into three sections. Between 1 sec. and 0.005 sec. and from 2 hr. to 5 min. the yield strength changed rapidly while the remainder of the curve from 5 min. to 1 sec. was practically without slope.

The glass fabric laminate was affected least by the rate of loading. The yield strength increased gradually over the time range for a 15 per cent gain. The behavior of the ultimate strength was somewhat erratic, dropping from an average of 54,000 psi. to 45,000 psi. in the time range from 5 min. to 2 hr. and having a wide scatter of from 61,000 psi. to 74,000 psi. in loading times of 0.5 to 1 sec. The remainder of the data from 5 min. to 0.005 sec. indicated an increase in ultimate strength of 10 per cent. The sudden changes in ultimate strength with speed of loading might be traced to the construction of the sheet which is composed of materials of widely varying properties. Since the glass offers very little opportunity for impregnation by the resin, it is possible the individual materials each have predominant effect at different loads, and this individual action is emphasized at different loading speeds.

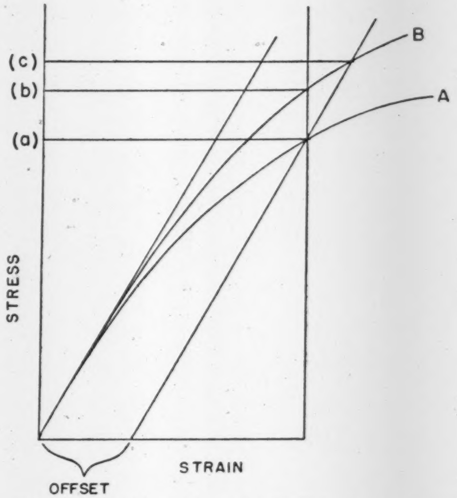


FIG. 12.—Difference in Yield Strength as Computed by Offset and Fixed Total Strain Methods.

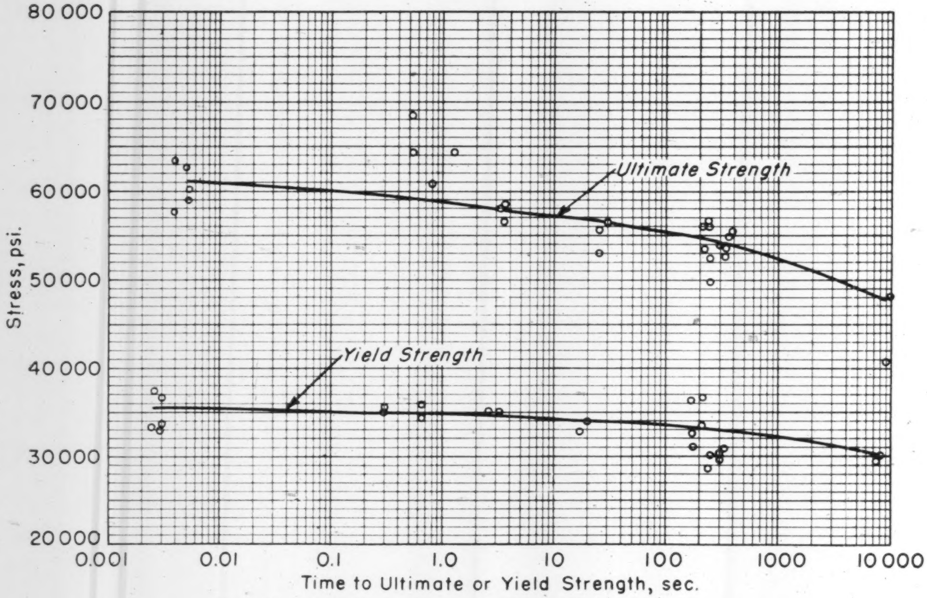


FIG. 13.—Stress *versus* Time—Glass Fabric Laminate.

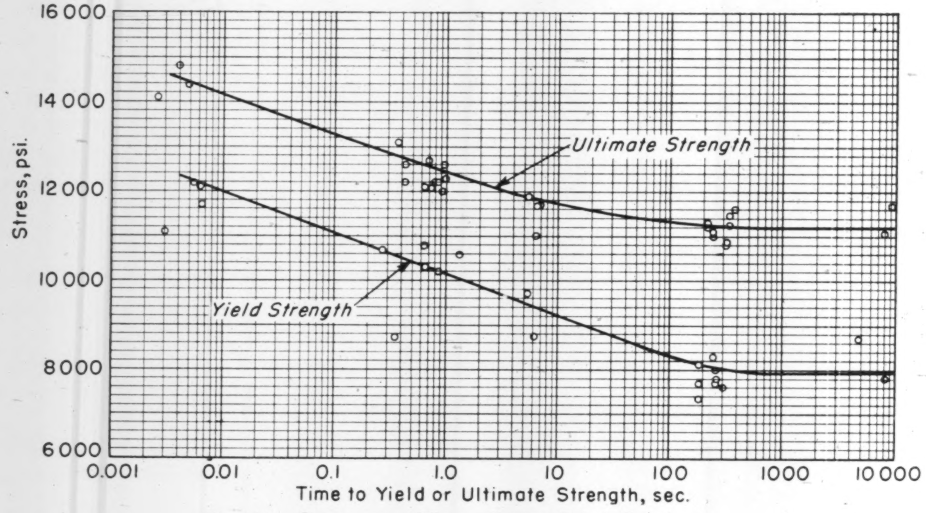


FIG. 14.—Stress *versus* Time—Cotton Fabric Laminate.

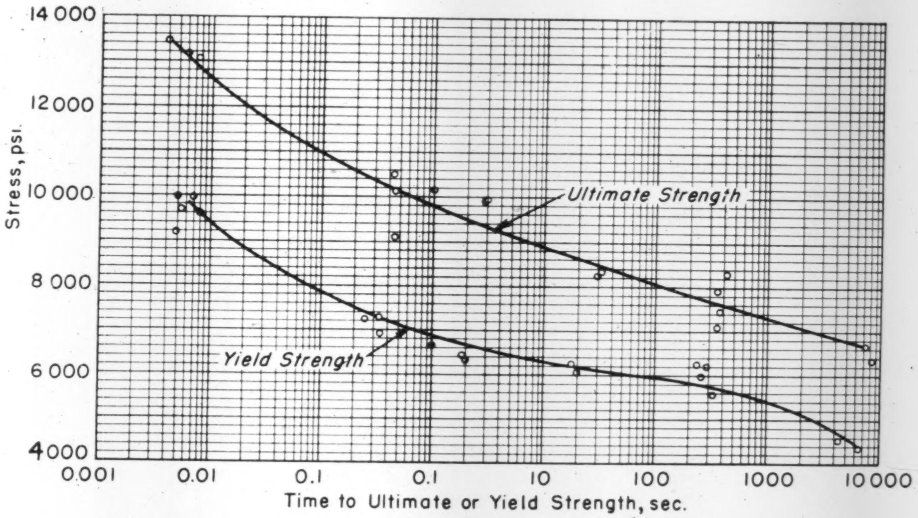


FIG. 15.—Stress *versus* Time—Methyl-methacrylate.

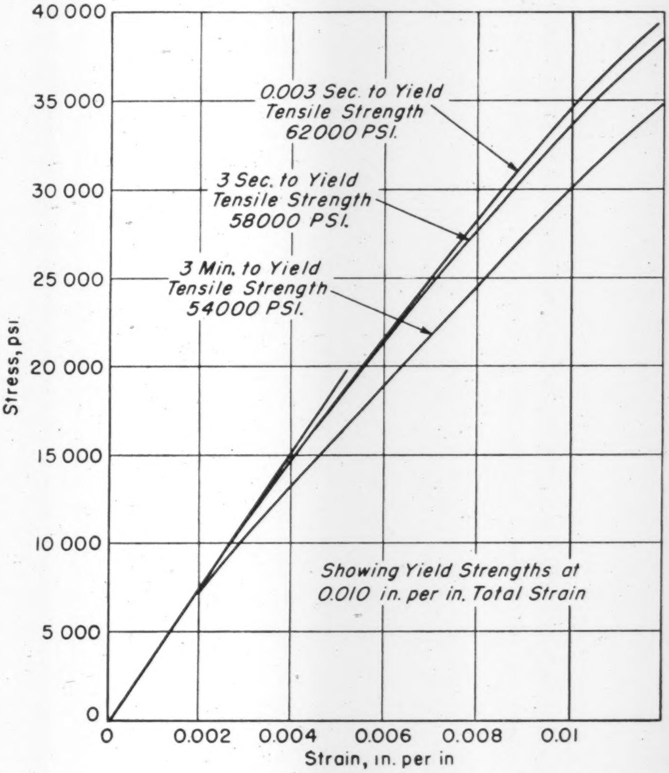


FIG. 16.—Typical Stress-Strain Curves of Glass Fabric Base Polyester Laminate at Different Loading Rates.

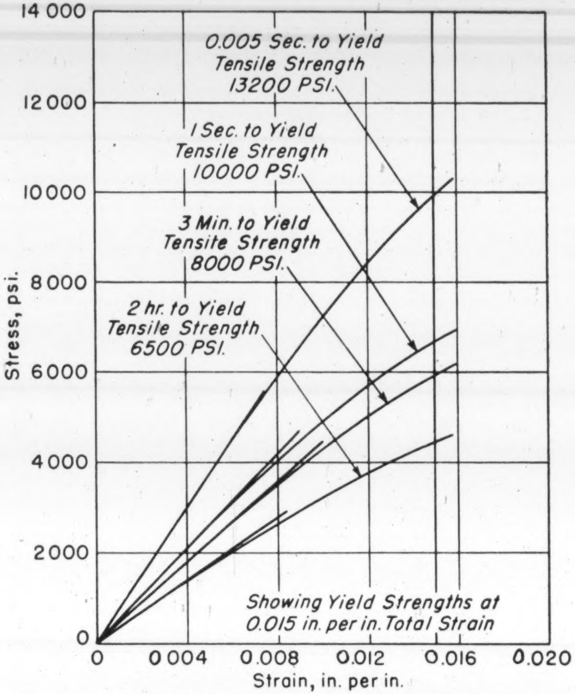


FIG. 17.—Typical Stress-Strain Curves of Polymethylmethacrylate at Different Loading Rates.

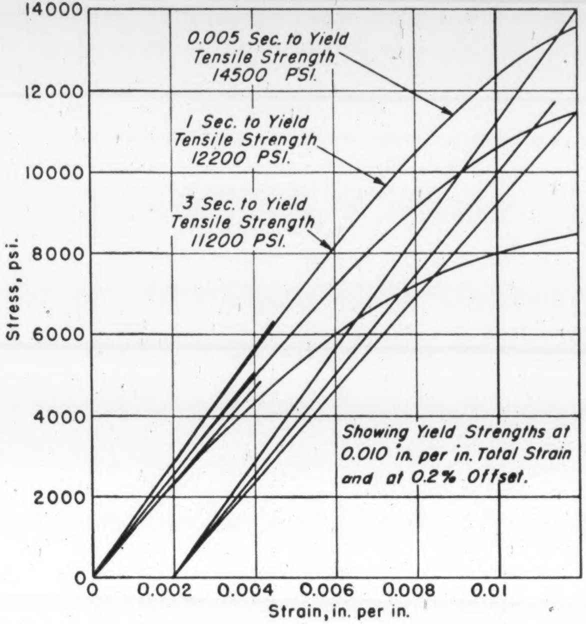


FIG. 18.—Typical Stress-Strain Curves of Cotton Fabric Base-Phenolic Laminate at Different Loading Rates.

The influence of rate of loading on the stress-strain curves of the non-metallic materials is represented by the typical curves at various rates of loading in Fig. 16, 17 and 18. These families of curves indicate that the glass fabric laminate is least affected by loading rate having a relatively constant modulus. All of the materials retain their characteristic stress-strain curves, but the cotton fabric laminate and methylmethacrylate curves are influenced, in their entirety, by the rate of loading, each developing greater moduli with increasing loading rates. The initial modulus of elasticity of the cotton fabric laminate increased approximately 20 per cent over the time range of 2 hr. to 0.005 sec. and that of the polymethylmethacrylate about 100 per cent for the same time range. There was no significant increase in initial modulus of elasticity over the range tested for the glass fabric base laminates. The more pronounced increase of modulus with loading rate of the latter material is explained by the plastoelastic behavior of the linear polymer, which provides a gradual flow of material at slow loading rates and a combination of viscous and elastic reactions at the higher rates of loading (7). The relationship between the curvature of the stress-strain curve and its sensitivity to loading rate is shown by the typical curves. Both the cotton fabric laminate and methylmethacrylate stress-strain diagrams have considerable curvature, and each is greatly affected by rate of loading while the glass laminate stress-strain curve is relatively straight and, as expected from its shape, is only slightly affected by rate of loading, (7).

TABLE V.—YIELD STRENGTH OF ALUMINUM ALLOY SHEET AT VARIOUS RATES OF LOADING.

Alloy[a]	Oscilloscope Record From C-1 SR-4 Strain Gage		Olsen Strain Gage
	Time, sec.[b]	Yield Strength, 0.2 per cent offset, psi.	Yield Strength, 0.2 per cent offset, psi.
24S-T3	0.62	47 800	
24S-T3	200		
24S-T3	0.003	52 500	52 600[c]
24S-T3			
75S-T6	0.90	72 200	
75S-T6	200		77 300[c]
75S-T6	0.006	76 000	

[a] 0.064 in. bare sheet.
[b] To yield strength.
[c] Static Loading.
Note.—All specimens taken in longitudinal direction.

The manner in which the yield strengths of 75S-T and 24S-T vary with rate of loading is indicated in Table V. Low stress values, obtained with SR-4 gages Type C-1, relative to control tests made with standard strain gages were obtained in these tests, probably because of an unbalanced bridge necessitated by a shortage of gages at the time, but the error was believed constant, so that relative effects of the speeds used are correct. The yield strength of the 75S-T aluminum alloy increased about 5 per cent over the loading time range of 0.90 sec. to 0.006 sec. and that for the 24S-T alloy 9 per cent over the loading time range of 0.62 sec. to 0.003 sec. No further tests were made on the aluminum alloys because of the small effect of rate of loading. Tests results on aluminum alloys and three steels are given in a paper by Clark and Wood (1) and also indicate no appreciable speed effect on aluminum alloys. Also additional information regarding tension impact tests of metals is given in previous papers (2, 3, 4, and 5).

CONCLUSIONS

Over the range of rates of loading investigated, the plastic materials exhibited the following changes in properties.

1. The yield strength, ultimate strength, and modulus of elasticity of the

methyl methacrylate each increased approximately 100 per cent.

2. The yield strength, ultimate strength, and modulus of elasticity of the cotton fabric laminate increased 45 per cent, 30 per cent, and 20 per cent respectively.

3. The yield strength and ultimate strength of the glass fabric laminate increased 15 per cent and 25 per cent, respectively, while the modulus of elasticity was relatively unaffected.

The yield strengths of the 75S-T and 24S-T aluminum alloys increased only slightly over the range of rates of loading investigated.

REFERENCES

(1) D. S. Clark and D. Wood, "Rapid Loading Properties of Aircraft Metals," *Technical Report No. 5742*, United States Air Force, Air Materiel Command. (to be published).

(2) D. S. Clark, "The Influence of Impact Velocity on the Tensile Characteristics of Some Aircraft Metals and Alloys," *Technical Note No. 868*, Nat. Adv. Committee of Aeronautics, October, 1942.

(3) E. R. Parker and C. Ferguson, "The Effect of Strain Rate Upon the Tensile Impact Strength of Some Metals," *Transactions*, Am. Soc. Metals, Vol. 30, p. 98 (1942).

(4) D. S. Clark and G. Datwyler, "Stress-Strain Relations Under Tension Impact Loading," *Proceedings*, Am. Soc. Testing Mat. Vol. 38, Part II, p. 98 (1938).

(5) P. E. Duwez, H. E. Martens, and D. S. Clark, "A Preliminary Study of the Influence of Rapid Loading and Time at Load on the Initiation of Plastic Deformation in Tension," *National Defense Research Committee Report No. M.450*, National Defense Research Committee, January, 1945.

(6) D. Rosato, "Acrylic Plastic Sheet Investigation of Tensile Properties at Low and Elevated Temperatures By Means of SR-4 Electric Strain Gages," *Bureau of Aeronautics Report No. AML NAM AE 440002*, United States Navy Department, April, 1947.

(7) Turner, Alfrey, Jr., "Mechanical Behavior of High Polymers," Vol. VI, pp. 186–189, Interscience Publishers, Inc., Lancaster, Pa. First Edition. (1948).

DISCUSSION

MR. D. S. CLARK[1] (*presented in written form*).—This investigation is of interest to those concerned with the properties of materials under conditions of dynamic loading, and of particular interest to those concerned with the properties of plastics.

The author states that his results on 24S-T and 75S-T aluminum alloys confirm the findings of Clark and Wood.[2] In comparing these results, it should be pointed out that the author's method of testing differs from that of Clark and Wood. Mr. Klinger's test involves the continuous application of load up to failure, while that of Clark and Wood involves the application of a specified load applied at a specified rate. Furthermore, the latter tests do not carry the specimens to failure. The time required to initiate plastic deformation is taken as the time interval beginning at the instant the constant stress is attained and ending when plastic strain begins. The agreement between the two tests in the case of the two aluminum alloys is good because of the properties of the material. However, it is expected that agreement might not be satisfactory with some materials such as an annealed low-carbon steel in which a definite yield point occurs.

The curves in Figs. 13, 14, and 15 are of an entirely different type than those reported by Clark and Wood and should not be confused.

These comments are not intended to detract from the work of Mr. Klinger but merely to point out a difference in the type of tests which lead to curves having similar appearance but with an entirely different meaning.

MR. D. A. SHINN[3] (*presented paper for author*).—I might say to Professor Clark for clarification, that the comparison made was based on the derivation of stress-strain curves from Professor Clark's stress-time and load-time curves, rather than on his delay time curves.

MR. S. A. GORDON.[4]—Several years ago I ran some tests similar to the ones discussed by the author, right through to failure. I can confirm his opinion, at least that there is no increase in yield strength, and none in the ultimate strength, with rates of loading down to about 0.35 both 24S-T and 75S-T.

MR. J. C. MILLSON.[5]—According to Mr. Klinger's introduction it would be "desirable to have an understanding of the relationship between rates of loading as encountered in aircraft and the stress-strain characteristics of the material." This is a very desirable motive but a number of authors have already shown that tensile strength of aluminum alloys is relatively unaffected by large increases of straining speed. Owing to the high ratio of 0.2 per cent offset yield strength to tensile strength for the alloys tested it can also be concluded that the yield strength is insensitive to changes in strain rates.

A strength property which might be

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[1] Associate Professor of Mechanical Engineering, California Institute of Technology, Pasadena, Calif.

[2] D. S. Clark and D. S. Wood, "The Time Delay for the Initiation of Plastic Deformation at Rapidly Applied Constant Stress," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 717 (1949).

[3] Materials Engineer, Air Materiel Command, Wright-Patterson Air Force Base, Dayton, Ohio.

[4] Chief Structural Test Engineer, The Glenn L. Martin Co., Baltimore, Md.

[5] Assistant Director of Research, Aluminium Laboratories, Limited, Kingston, Ontario, Canada.

appreciably influenced by speed of loading, namely, the proportional limit, appears to have been given no consideration. It is true that it is difficult to measure accurately and is relatively little used in design. On the other hand, the stress-strain curve shown in Fig. 11 of the paper offers proof that it has been affected and that it might also be measured. The sharp "knee" of this curve is not typical for normal 75S-T sheet and could be explained by an increase in the proportional limit.

Examination of a number of curves obtained in this laboratory leads us to believe that, although 75S-T has a relatively sharply breaking curve, only under special circumstances would it correspond to the one shown.

It is possible that the limitations of the instructions covering the testing program were such that the 0.2 per cent yield strength was of specific interest. On the other hand, although the proportional limit is difficult to measure and its use is limited as far as normal design is concerned, it is one property which might be expected to change appreciably with an increase in the rate of loading. Under such tests as the author made, it is unfortunate that it was not also given some thought in planning the work.

MR. SHINN.—To answer the question about Fig. 11, first I would say that was a fairly normal curve for an aluminum alloy.

To get back to the reason for the tests, an aircraft designer is very much interested in adding as much data as possible on stress strain under many conditions, in order that he can utilize all the information he can possibly obtain to design the best possible and the lightest possible aircraft.

The purpose of the test was to obtain this stress-strain data for him rather than any anticipation that we would find any particularly large increase in the yield strength. It is the same sort of reason for which you would obtain stress-strain curves at various temperatures.

MESSRS. STEVEN YURENKA[6] AND ALBERT G. H. DIETZ[7] (*by letter*).—In the plastics laboratory at the Massachusetts Institute of Technology we have tested polymethylmethacrylate at various constant rates of strain and constant rates of load and have also verified that the strength of this material is as sensitive to loading rates as given by the author in Table III and in Figs. 15 and 17. Although over a smaller range of times to reach ultimate strength; that is, from 0.5 to 1000 sec., near straight-line relationships between time and strength of polymethyl methacrylate similar to those of Fig. 15 were also obtained.

With regard to crazing of test specimens when "Duco" cement was used to glue strain gages to polymethylmethacrylate, we have found, after trying a large number of cements, that for paper-backed gages 10 per cent carbon tetrachloride, 90 per cent solution of 10 per cent polymer in monomer catalyzed with 0.5 per cent benzoyl peroxide yielded good bonds without crazing.

Although, as the author pointed out, no precautions were taken to compensate for temperature changes during tests because test durations were short, would it not have been better to account for the effect of these changes between calibration and actual testing?

Instead of using an average value of strain for the calibrating resistance because of the curvature of the tube which produces a 22 per cent variation in displacement for a given signal, it is possible to correct this situation by a simple electrical device. If a stepwise calibrating signal to provide x and y coordinates is introduced in a manner similar to that in which data are fed to the recorder, the optical (photographic) errors as well as electrical errors in linearity can be compensated.

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[6] Research Associate, Plastics Laboratory, Department of Building Engineering and Construction, Massachusetts Institute of Technology, Cambridge, Mass.

[7] Professor, Department of Building Engineering and Construction, Director, Plastics Laboratory, Massachusetts Institute of Technology, Cambridge, Mass.

A horizontal, x, sweep can be provided while vertical y steps are taking place so that a series of 20 horizontal, x, lines, spaced linearly by the calibrator in a vertical, y, direction appear on the cathode ray tube. A similar vertical, y, sweep is applied while calibrating horizontally, x. This grid is photographed first and then the test started with the dot located at the proper corner of the oscilloscope screen. Since the curvature of the tube produces the same variation in grid lines and test lines alike, nonlinearities from this effect are thereby avoided.

MR. R. F. KLINGER (*author's closure*).—The author wishes to thank Messers. Clark, Gordan, Millson, Yurenka and Dietz for the discussions and suggestions presented.

Mr. Shinn has satisfactorily clarified the points of question brought out by Clark and Millson. A study of the proportional limit as stressed by Mr. Millson would necessitate an expansion of that particular section of the stress-strain curve and, of course, eliminate for study the remainder of the stress-strain relations at other stages.

It is gratifying to find partial substantiation of results as provided by Yurenka and Dietz inasmuch as the strength of the polymethylmethacrylate appeared to be so drastically dependent on the rate of straining. In answer to the question of variation of calibration with temperature change, it can be said that within the precision of the test method the calibration did not change over a period of days as evidenced by periodic recalibrations. As during the loading, the time between placement of the calibration lines and application of load was relatively short, thus, preventing temperature changes of importance. The method of calibration as suggested by Yurenka and Dietz would have been quite helpful by eliminating the errors as specified. The original intention was, of course, to maintain the equipment and indications as simple and direct as possible, thereby eliminating any errors which might be induced by the personal analysis of a more complex system. The calibration method used provided satisfactory correlation with a standard measurement method as indicated in the paper.

COMBINED TENSION-TORSION CREEP-TIME RELATIONS FOR ALUMINUM ALLOY 2S-0*

BY JOSEPH MARIN,[1] J. H. FAUPEL[2], AND L. W. HU[3]

SYNOPSIS

Many machine and structural parts are subjected to creep and a combined state of stress. Most experimental investigations on the creep properties of materials, however, have been made under conditions of simple stress. There have been only a few investigations reported on the creep-time relations of materials subjected to combined stresses (**1**, **2**, **3**, **4**).[4] This paper gives the results of an investigation on the combined stress creep properties of Alcoa 2S-O. Combined states of stress were produced by subjecting thin-walled tubular specimens of circular cross-section to various combinations of axial tension and torsion. The minimum constant creep rates for various values of the stresses and for ratios of the biaxial principal stress from 0 to −1.0 were found to be in approximate agreement with values predicted theoretically using simple tension creep test results.

COMBINED TENSION-TORSION CREEP TEST RESULTS

Material Tested:

The material tested was an aluminum alloy 2S-O. This material is commercially pure aluminum in the fully annealed condition. The material was supplied in tubular extruded form in lengths of 16 ft., with an internal diameter of 1 in. and a wall thickness of $\frac{1}{4}$ in. The mechanical properties of the material, as supplied by the Aluminum Company of America, are as follows: tensile strength = 13,000 psi., yield strength (0.2 per cent offset) = 5000 psi., modulus of elasticity = 10×10^6 psi., elongation in 2 in. = 35 to 45 per cent, and Poisson's ratio = 0.33.

Test Specimen:

The biaxial stress specimens were machined to the dimensions shown in Fig. 1. The specimen used had an over-all length of 7 in. with an intermediate length of 3.75 in. of reduced wall thickness equal to approximately 0.10 in. The wall thickness was determined at 48 places on each specimen to 0.0001 in. by using the wall measuring equipment described by Marin *et al.* (**5**). The wall thickness - diameter ratio for the specimens was approximately 1:10 so that the biaxial stresses throughout the wall were essentially constant. A smaller wall thickness ratio could not be used since it was found by test that failures by buckling would occur.

Testing Machine:

A new type of combined tension-torsion machine was designed and constructed for this project. In this machine (Fig. 2), a tubular specimen, *S*, can be subjected simultaneously to tension and

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Professor of Engineering Mechanics, The Pennsylvania State College, State College, Pa.

[2] Research Engineer, Materials of Construction Section, E. I. du Pont de Nemours and Co., Inc., Wilmington, Del. Formerly Research Assistant and du Pont Fellow in Engineering Mechanics at The Pennsylvania State College.

[3] Research Assistant, The Pennsylvania State College, State College, Pa.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1070.

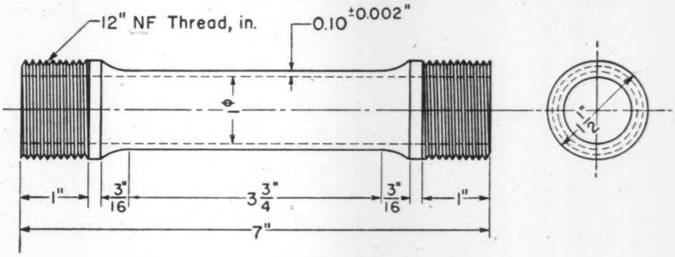

12" NF Thread, in.
$0.10^{\pm 0.002"}$
1"
$\frac{3"}{16}$
$3\frac{3"}{4}$
$\frac{3"}{16}$
1"
7"
$1\frac{1"}{2}$

FIG. 1.—Combined Tension-Torsion Specimen.

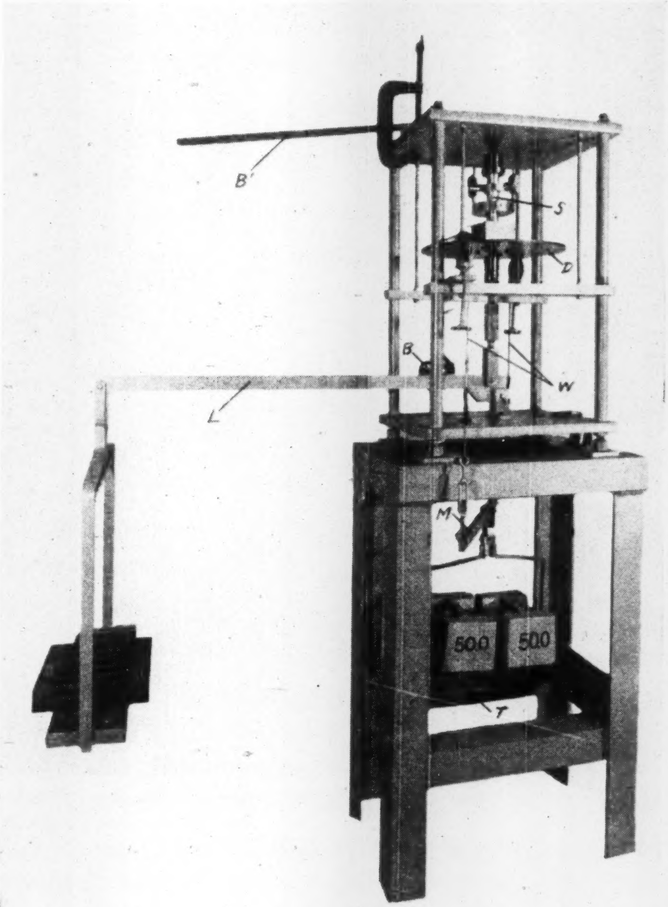

B'
S
D
B
W
L
M
50.0
50.0
T

FIG. 2.—Combined Tension-Torsion Creep Machine.

torsional loads. The axial tensile load is applied by a lever, *L*, to which weights are attached as shown in Fig. 2. The lever ratio for the tensile load is 40:1. After a predetermined tensile load is applied to the end of lever, *L*, the lever is adjusted to a horizontal position by turning a compensator and by the use of a level bulb, *B*, mounted on the top side of the lever.

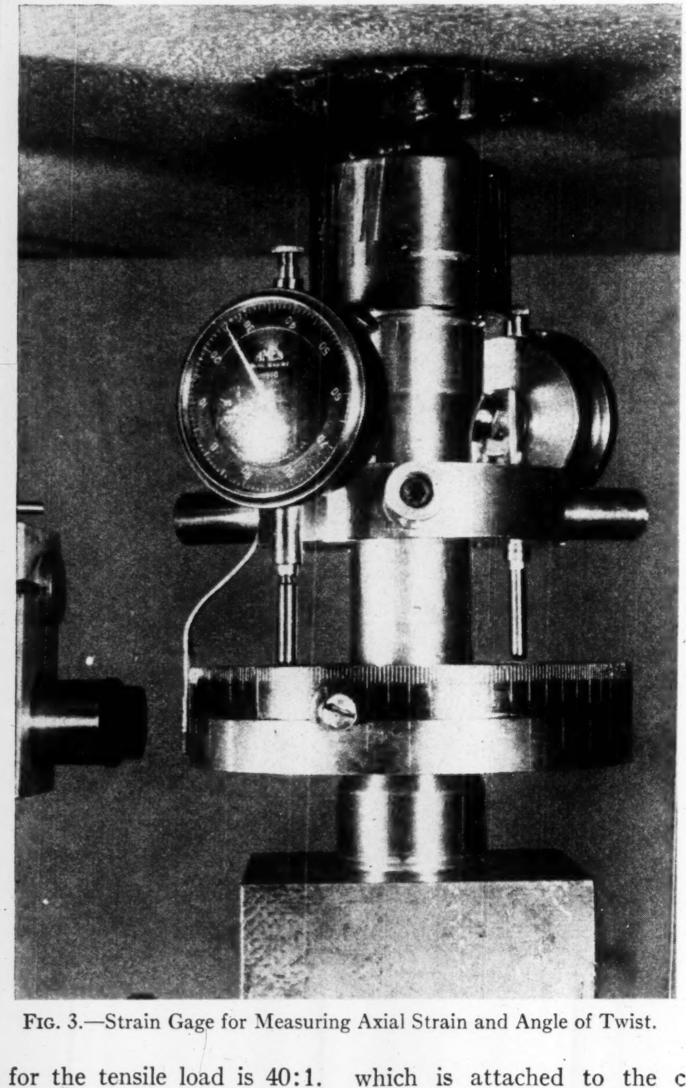


FIG. 3.—Strain Gage for Measuring Axial Strain and Angle of Twist.

The torsion load is applied by cables, *W*, attached to the torsion disk, *D*. Weights applied to the cable bar, *M*, which is attached to the cables, *W*, applying the torsion loads to the disk, *D*. The torsion disk is attached to the specimen, *S*. As the specimen twists through large angles the cables unwind around the disk and the weights on the torsion pan, *T*, are lowered. A regulating bar, B', on top of the machine is used to turn the specimen and thereby raise the weights and pan.

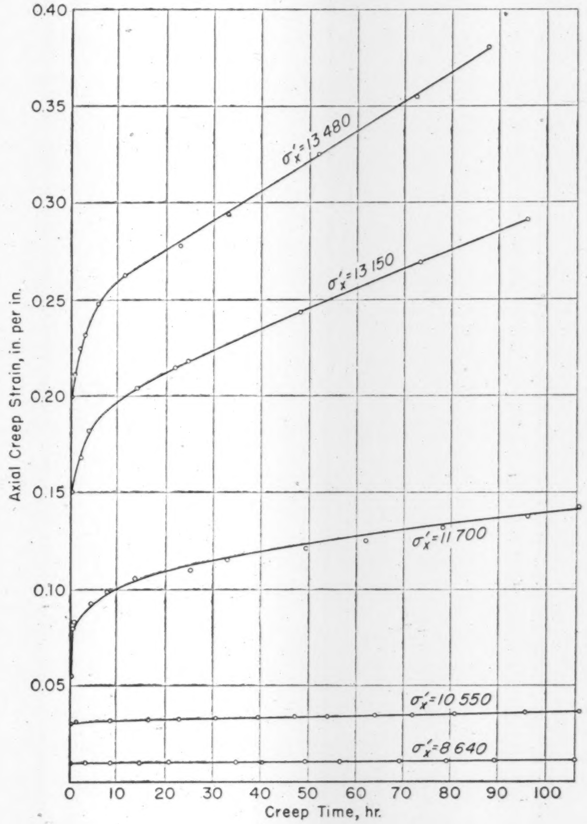


Fig. 4.—Creep-Time Relations for Simple Tension Tests.

Measurement of Strains:

A special strain gage (Fig. 3) was designed and constructed for measuring large axial strains and angles of twist. The axial tensile strains were measured over a 2-in. gage length by two 0.0001-in. dial gages placed 180 deg. apart as shown in Fig. 3. The angles of twist were measured directly as shown in Fig. 3 for a 2-in. gage length and to the nearest $\frac{1}{4}$ deg. Large axial strains and angles of twist could be measured by preventing the rings from slipping relative to the specimen. The slipping is prevented by three radial rods with concealed ends attached to the rings and preloaded with helical springs. The preload in the springs is maintained throughout the test as the specimen diameter decreases due to the axial tensile loads.

Test Procedure:

For a selected ratio of the nominal principal stresses, the required values of the axial tensile load and torque load were calculated. The tensile load was

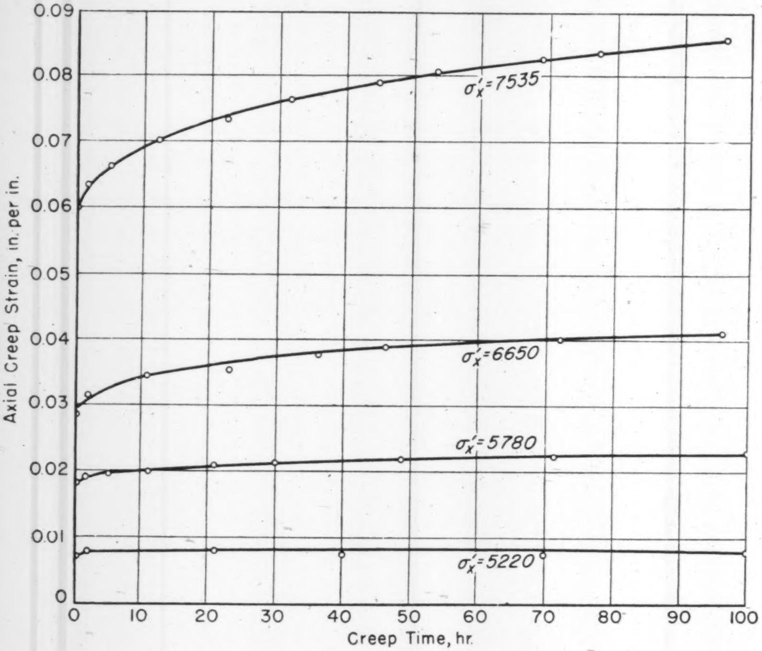


FIG. 5.—Creep-Time Relations for Tension-Torsion Tests (Axial Strain). Stress Ratio: $\sigma_2/\sigma_1 = -0.33$.

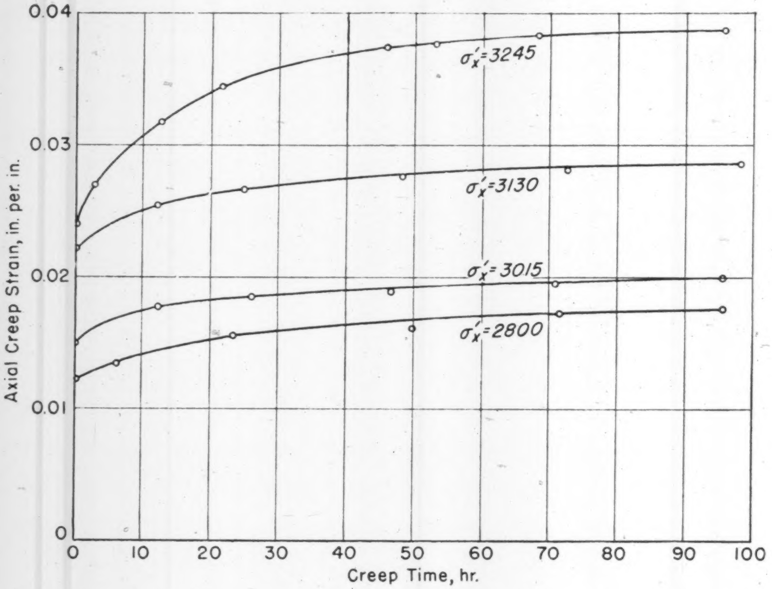


FIG. 6.—Creep-Time Relations for Combined Tension-Torsion Tests (Axial Strain). Stress Ratio: $\sigma_2/\sigma_1 = -0.66$.

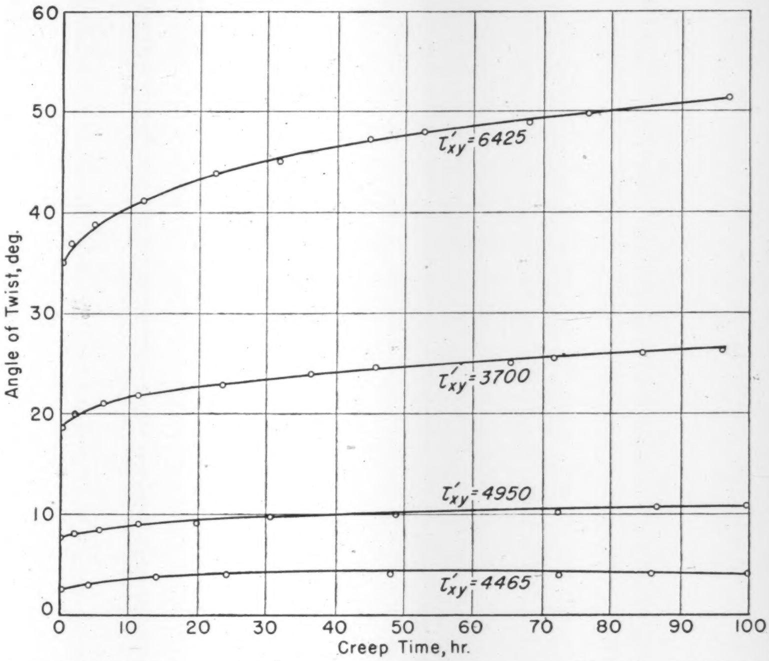


FIG. 7.—Creep-Time Relations for Combined Tension-Torsion Tests (Shear Strains). Stress Ratio: $\sigma_2/\sigma_1 = -0.33$.

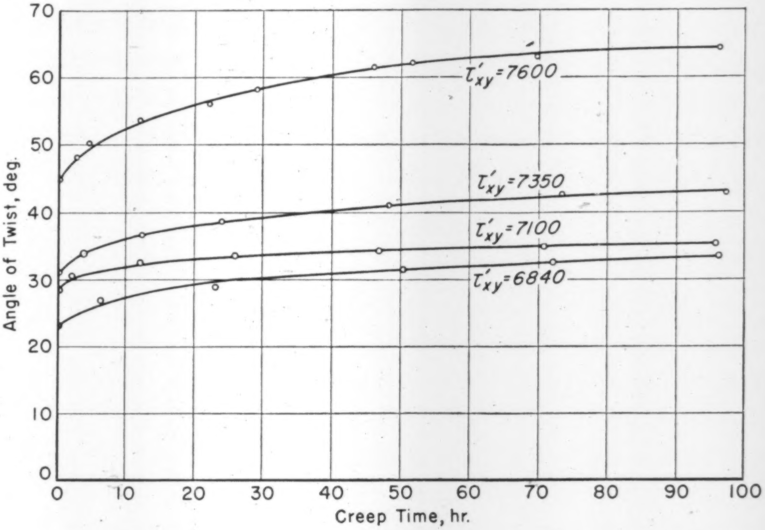


FIG. 8.—Creep-Time Relations for Combined Tension-Torsion Tests (Shear Strains). Stress Ratio: $\sigma_2/\sigma_1 = -0.66$.

then first applied followed immediately by the torque load. Axial strain and angle of twist readings were then recorded for predetermined intervals of time up to 100 hr. In all cases, the initial creep strains were recorded in less than 5 min. from the time of application of the load. In selecting the load values for each principal stress ratio, the lowest loads used were close to those producing yielding of the material, while the highest loads used were close to those values which would produce failure by buckling. At least four tests were conducted for each principal stress ratio, and the nominal principal stress ratios used were 0 (simple tension), -0.33, -0.66 and -1.0 (pure torsion).

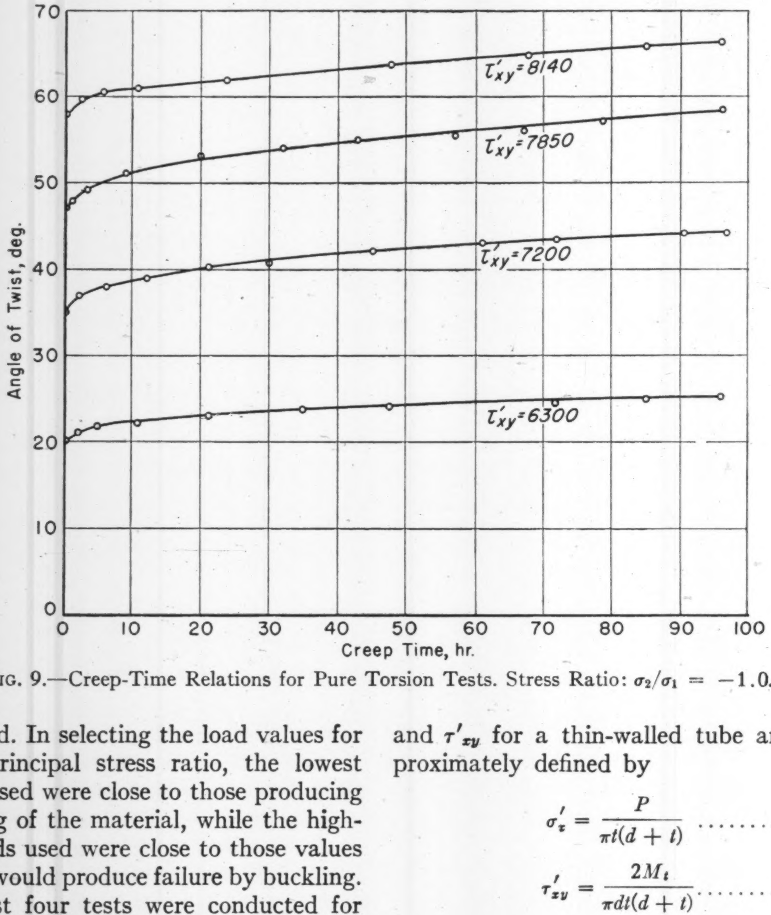


FIG. 9.—Creep-Time Relations for Pure Torsion Tests. Stress Ratio: $\sigma_2/\sigma_1 = -1.0$.

The nominal principal stress ratio is

$$R = \sigma_2/\sigma_1 = \frac{\sigma'_x/2 - \sqrt{(\sigma'_x/2)^2 + (\tau'_{xy})^2}}{\sigma'_x/2 + \sqrt{(\sigma'_x/2)^2 + (\tau'_{xy})^2}} \quad \ldots (1)$$

where:
σ'_x = the nominal axial stress, and
τ'_{xy} = the nominal shear stress.

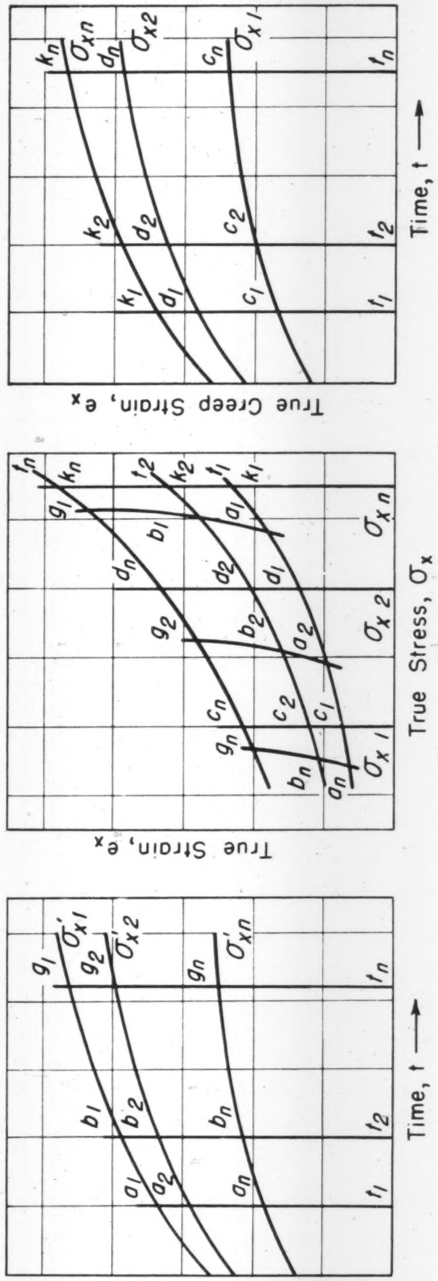
If P = the axial load, M_t = the twisting moment, d = the internal diameter and t = the wall thickness, then the values of the nominal stress components σ'_x and τ'_{xy} for a thin-walled tube are approximately defined by

$$\sigma'_x = \frac{P}{\pi t(d + t)} \quad \ldots\ldots\ldots\ldots (2)$$

$$\tau'_{xy} = \frac{2M_t}{\pi d t(d + t)} \quad \ldots\ldots\ldots\ldots (3)$$

Placing values of σ'_x and τ'_{xy} from Eq. 2 and 3 in Eq. 1, the nominal principal stress ratio is

$$R = \frac{1 - \sqrt{1 + \left(\frac{4M_t}{Pd}\right)^2}}{1 + \sqrt{1 + \left(\frac{4M_t}{Pd}\right)^2}} \quad \ldots\ldots (4)$$


Nominal Creep Strain, e_x
Time, t ⟶
a_1
a_2
a_n
b_1
b_2
b_n
g_1
g_2
g_n
σ'_{x1}
σ'_{x2}
σ'_{xn}
t_1
t_2
t_n
True Strain, e_x
True Stress, σ_x
σ_{x1}
σ_{x2}
σ_{xn}
a_1
a_2
a_n
b_1
b_2
b_n
c_1
c_2
c_n
d_1
d_2
d_n
g_1
g_2
g_n
k_1
k_2
k_n
t_1
t_2
t_n
True Creep Strain, e_x
Time, t ⟶
c_1
c_2
c_n
d_1
d_2
d_n
k_1
k_2
k_n
σ_{x1}
σ_{x2}
σ_{xn}
t_1
t_2
t_n

(a)

(b)

(c)

FIG. 10.—Determination of True Creep-Time Relations.

Creep Test Results:

For the combined stress tests the unit creep strains are the nominal axial creep strain e_x and the nominal shear creep strain γ_{xy}. The values of these creep strains in terms of the measured deformations are

$$e_x = \frac{\delta_{1x} + \delta_{2x}}{2L_o} \quad \ldots\ldots\ldots\ldots (5)$$

and

$$\gamma_{xy} = \frac{\pi}{720}(d + 2t)\rho \quad \ldots\ldots\ldots (6)$$

where:

δ_{1x}, δ_{2x} = the measured axial strains on two opposite sides of the specimens,

L_o = the original gage length, and

ρ = the angle of twist in degrees.

By Eqs. 5 and 6, the unit nominal creep strains were determined.

Figures 4 to 9 inclusive give the nominal creep strain-time relations for the various principal stress ratios and for both the axial strain e_x and the shear strain γ_{xy}. For the principal stress ratio of zero, that is, for simple tension, only one graph showing the axial creep strain is obtained as shown in Fig. 4 Similarly, for pure torsion or for the principal stress ratio of −1.0, only the shear creep strain-time relations can be obtained as shown in Fig. 9. For intermediate values of the principal stress ratio or for $R = -0.33$ and -0.66, the axial and shear creep-time relations are given, as shown in Figs. 5, 6, 7, and 8.

Lack of time and equipment made it necessary to limit the tests to 100 hr. as represented in Figs. 4 to 9.

INTERPRETATION OF CREEP TEST RESULTS

Methods of interpretation of combined creep test results based upon simple tension creep data have been discussed by

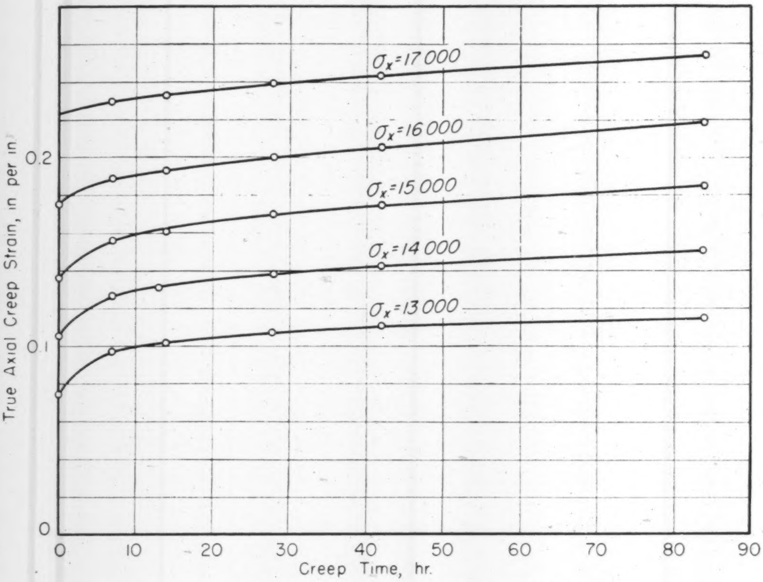


FIG. 11.—True Axial Stress, Creep-Time Relations for Simple Tension.

several investigators (1, 6, 7, 8). The limited amount of test data available on combined creep stresses (1, 8) indicates that the theory, which will be called the Modified St. Venant Theory, appears to be a good approximation for predicting combined stress creep strains. This theory is derived and discussed by Soderberg (6) and Marin (8). At the outset, one of the difficulties encountered in applying the St. Venant Theory is the fact that the theory assumes small strains. The large strains produced in the test reported, however, result in considerable reduction in the cross-sectional areas of the specimens during the time covered by the test. With reduction in the cross-sectional area, the stresses cannot be considered as constant throughout a creep test. In other words, the creep tests are constant load tests and not constant stress tests. The foregoing statement does not apply for the pure torsion creep tests, since in these tests there is no change in the cross-sectional area. Furthermore, with large axial creep strains the nominal axial strain based on the original gage length, as defined by Eq. 5, is no longer correct.

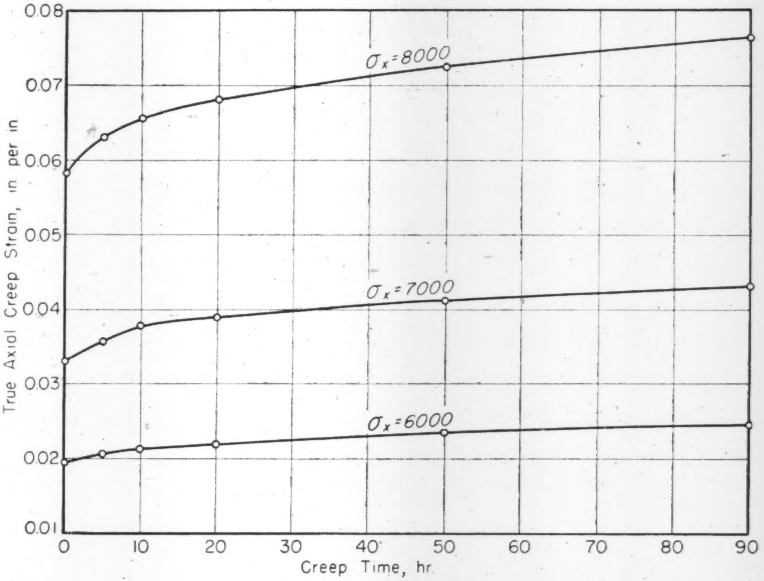


FIG. 12.—True Axial Stress, Creep-Time Relations for Nominal Combined Stress Ratio, −0.33.

To remedy the foregoing discrepancies, the true stresses and true creep strains based upon a changing cross-sectional area and changing gage length will first be determined. This can be done by first finding expressions for the diameter, d_p, and wall thickness, t_p, corresponding to any stage during the creep test. The true strain, ϵ, corresponding to a nominal strain, e_o, is obtained by noting that the true strain is

$$\epsilon = \int_{L_o}^{L} \frac{dL}{L} = ln\,\frac{L}{L_o} = ln\,\frac{L_o + \Delta L}{L_o}$$

or

$$\epsilon = ln\,(1 + e_o) \ldots\ldots\ldots\ldots (7)$$

By Eq. 7 the true strain in the direction of the wall thickness is

$$\epsilon_z = ln\,(1 + e_z) = ln\,\frac{t_p}{t} \qquad \text{........(8)}$$

If it is assumed that the volume remains constant, $\epsilon_x + \epsilon_y + \epsilon_z = 0$, and since $\sigma_z = 0$ and $\sigma_y = 0$, then $\epsilon_y = \epsilon_z$ and $2\epsilon_z + \epsilon_x = 0$, or

$$\epsilon_z = -\frac{\epsilon_x}{2} = -\tfrac{1}{2}\,ln\,(1 + e_x) \qquad \text{......(9)}$$

By Eqs. 8 and 9 the wall thickness, t_p, in terms of the original wall thickness, t, and the nominal strain, e_x, becomes

$$t_p = t/(1 + e_x)^{1/2} \qquad \text{..........(10)}$$

The true diameter, d_p, at any time, t, is defined by

$$\epsilon_y = ln\,d_p/d \qquad \text{............(11)}$$

By Eqs. 9 and 11, since $-\frac{1}{2}\epsilon_x = \epsilon_y$, the diameter, d_p, at any time is defined by

$$d_p = d/(1 + e_x)^{1/2} \qquad \text{........(12)}$$

Replacing t and d in Eqs. 2 and 3 by t_p and d_p defined by Eqs. 10 and 12 the true stress components, σ_x and τ_{xy}, are

$$\sigma_x = \frac{P}{\pi t(d + t)}(1 + e_x) = \sigma_x'(1 + e_x) \qquad (13)$$

$$t_{xy} = \frac{2M_t}{\pi dt(d + t)}(1 + e_x)^{3/2} = \tau_{xy}'(1 + e_x)^{3/2} \qquad (14)$$

Equation 13 defines the true stress components, σ_x and τ_{xy}, in terms of the nominal stresses, σ'_x and τ'_{xy}, and the nominal axial strain e_x. By Eq. 7 the true axial strain becomes

$$\epsilon_x = ln\,(1 + e_x) \qquad \text{..........(15)}$$

For large shear strains the angle and the tangent of the angle cannot be assumed equal. The shear strain for large angles of twist, therefore, must be taken as

$$\gamma_{xy}' = \tan\,\gamma_{xy} = \tan\left[\frac{\pi}{720}(d + 2t)\rho\right] \qquad \text{..(16)}$$

For the creep angles of twist measured in this investigation, the values of γ'_{xy} and γ_{xy} are approximately equal. By Eqs. 13, 14, 15, and 16 the values of the true stresses and strains can be computed for any point on a creep-time

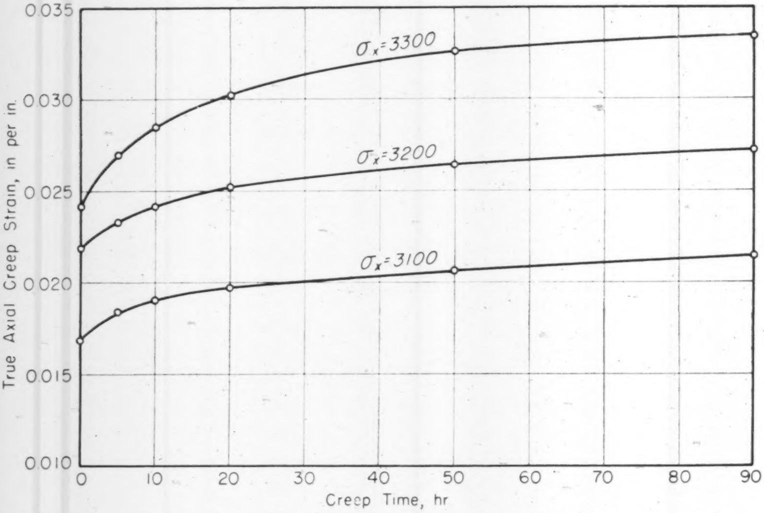


FIG. 13.—True Axial Stress, Creep-Time Relations for Nominal Combined Stress Ratio, −0.66.

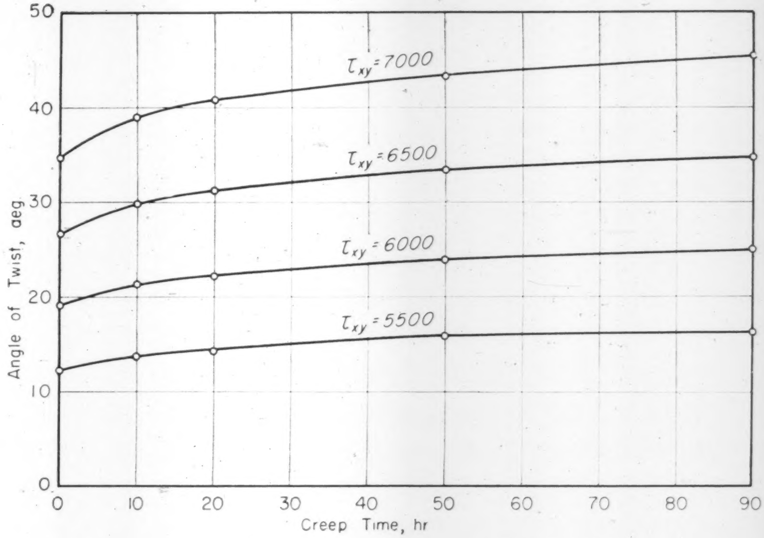


FIG. 14.—True Shear Stress, Creep-Time Relations for Nominal Combined Stress Ratio, −0.33.

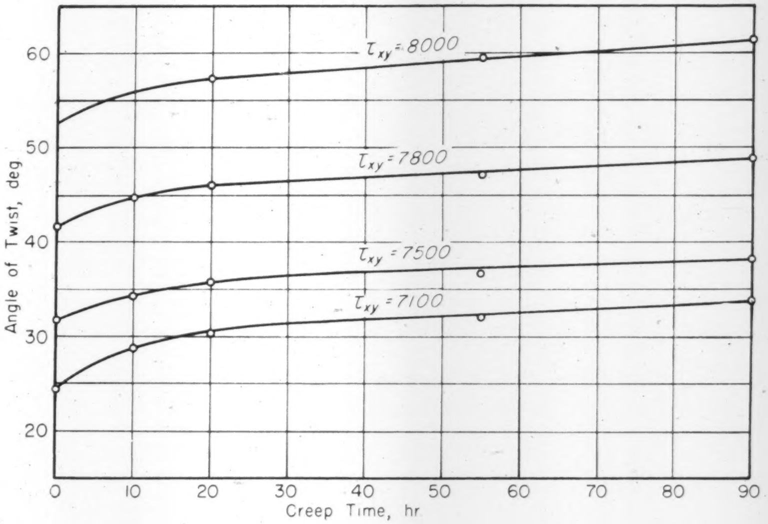


FIG. 15.—True Shear Stress, Creep-Time Relations for Nominal Combined Stress Ratio, −0.66.

curve. It is now possible to obtain approximate true creep-time relations for constant true stress values as outlined in the following.

Determination of True Creep-time Relations

A method of interpolation of test data is developed in the following to obtain true creep strain-time relations for constant true stress values from the moment creep-time curves for constant tensile loads. The procedure used can be explained by referring to Fig. 10.

Figure 10(a) represents nominal creep-time relations corresponding to a series of nominal stresses $\sigma'_{x1}, \sigma'_{x2} \ldots\ldots\ldots \sigma'_{xn}$. To obtain the true creep strain-time relations in Fig. 10(c) from the nominal creep strain-time relations of Fig. 10(a), the following steps are applied:

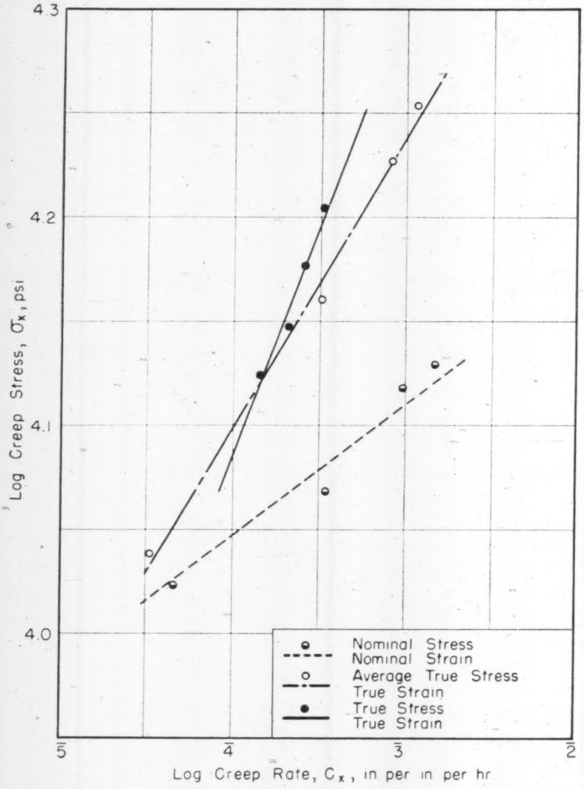


FIG. 16.—Log Creep Rate-Log Stress Relation for Simple Tension.

1. For selected times $t_1, t_2 \ldots\ldots\ldots t_n$ in Fig. 10(a), determine the true strains, ϵ_x, and true stresses, σ_x, using Eqs. 13 and 15 for points $a_1, a_2 \ldots\ldots\ldots a_n, b_1, b_2 \ldots\ldots\ldots b_n$, and $g_1, g_2 \ldots\ldots\ldots g_n$.

2. With values of true creep strains and true creep stresses determined in step 1, plot true creep stress-strain relations as shown in Fig. 10(b) for various times $t_1, t_2 \ldots\ldots\ldots t_n$ as parameters. Points a, b, and g in Figs. 10(a) and 10(b) are corresponding points.

3. For selected values of the true stresses $\sigma_{x1}, \sigma_{x2} \ldots\ldots\ldots \sigma_{xn}$ (Fig. 10(b)), determine creep strains $c_1, c_2 \ldots\ldots\ldots c_n$. $d_1, d_2 \ldots\ldots\ldots d_n, k_1, k_2 \ldots\ldots\ldots k_n$ corresponding to times $t_1, t_2 \ldots\ldots\ldots t_n$.

4. With values of the true creep strains obtained in step 3, true creep-time curves can be obtained as shown in Fig. 10(c). Points c, d, and k in Fig. 10(c) correspond to points c, d and k in Fig. 10(b).

Using Eqs. 14 and 16 for the true

shear stress and creep strain, the true shear creep-time relations can be obtained.

Applying the foregoing procedure, the true creep-time curves shown in Figs. 11 to 15 were obtained for the axial and shear strains and for the various principal stress ratios.

TABLE I.—VALUES OF CONSTANTS B AND n FOR CREEP IN SIMPLE TORSION.

Method Using	B	n
Nominal stress and nominal creep strain	3.51×10^{-53}	12.0
True stress and true creep strain	7.73×10^{-22}	4.2
Average true stress and true creep strain[a]	1.61×10^{-34}	7.3

[a] Method discussed in Appendix.

TABLE II.—COMPARISON OF EXPERIMENTAL AND THEORETICAL CREEP RATES.

Nominal Stress Ratio, $R = \sigma_2/\sigma_1$	True Axial Stress, σ_x, psi.	Axial Creep Rate, C_x			True Shear Stress, τ_{xy}, psi.	Circumferential Creep Rate, C_c		
		Experimental, in. per in. per hr.	Theoretical, in. per in. per hr.	Difference, per cent		Experimental, in. per in. per hr.	Theoretical, in per in. per hr.	Difference, per cent
−0.33	6000	3.86×10^{-5}	3.96×10^{-5}	−2	5500	14.3×10^{-5}	12.4×10^{-5}	15
	7000	6.15	7.58	−19	6000	18.4	17.9	3
	8000	12.0	13.2	−9	7000	31.3	34.2	−8
−0.66	3100	2.43	3.96	−41	7500	18.7	27.3	−31
	3200	2.94	4.52	−37	7800	21.9	32.4	−32
	3300	4.57	5.12	−12	8000	32.8	36.2	−9
−1.0	...	...	...	...	6300	13.09	12.14	8
	...	...	...	...	7200	24.33	21.36	14
	...	...	...	...	7850	34.8	30.71	13
	...	...	...	...	8140	35.0	35.7	−2

The shear creep-time relation for pure torsion shown in Fig. 9 also applies for the true shear creep-time relation since the angles of twist measured are of such magnitudes that $\gamma'_{xy} = \gamma_{xy}$ in Eq. 16.

Comparison of Theoretical and Experimental Creep Rates for Combined Stresses:

The Modified St. Venant Theory (6, 8) for predicting creep rates for combined stresses based on tension creep test results will be used in the following interpretation. To apply the theory it is first necessary to evaluate a creep rate-stress relation for creep in simple tension.

An examination of the tension creep test results shows that the creep rate at 100 hr. is approximately related to the stress by the equation

$$C = B\sigma^n \quad \ldots\ldots\ldots\ldots (17)$$

where B and n are experimental constants. By Eq. 17 a straight-line relation exists between the *log C* and *log σ*. Figure 16 gives a plot of the *log C versus log σ* for both the nominal stress and creep strain data obtained from Fig. 4 and the true stress and creep strain data as given in Fig. 11. Values of B and n for both the nominal and true values are listed in Table I.

Using the true stress and true creep strain constants, Eq. 17 becomes

$$C = 7.73 \times 10^{-22}\sigma^{4.2} \quad \ldots\ldots (17a)$$

By the Modified St. Venant Theory (6, 8) it can be shown that the axial and circumferential creep rates in terms of the true axial tensile stress σ_x and the true shear stress τ_{xy} are

$$C_x = B(\sigma_x^2 + 3\tau_{xy}^2)^{(n-1)/2}(\sigma_x) \quad \ldots\ldots (18)$$

$$C_c = 3B(\sigma_x^2 + 3\tau_{xy}^2)^{(n-1)}/2(\tau_{xy}) \quad \ldots (19)$$

Placing values of B and n based on the true creep strain-stress data in Eqs. 18 and 19, the theoretical or predicted axial

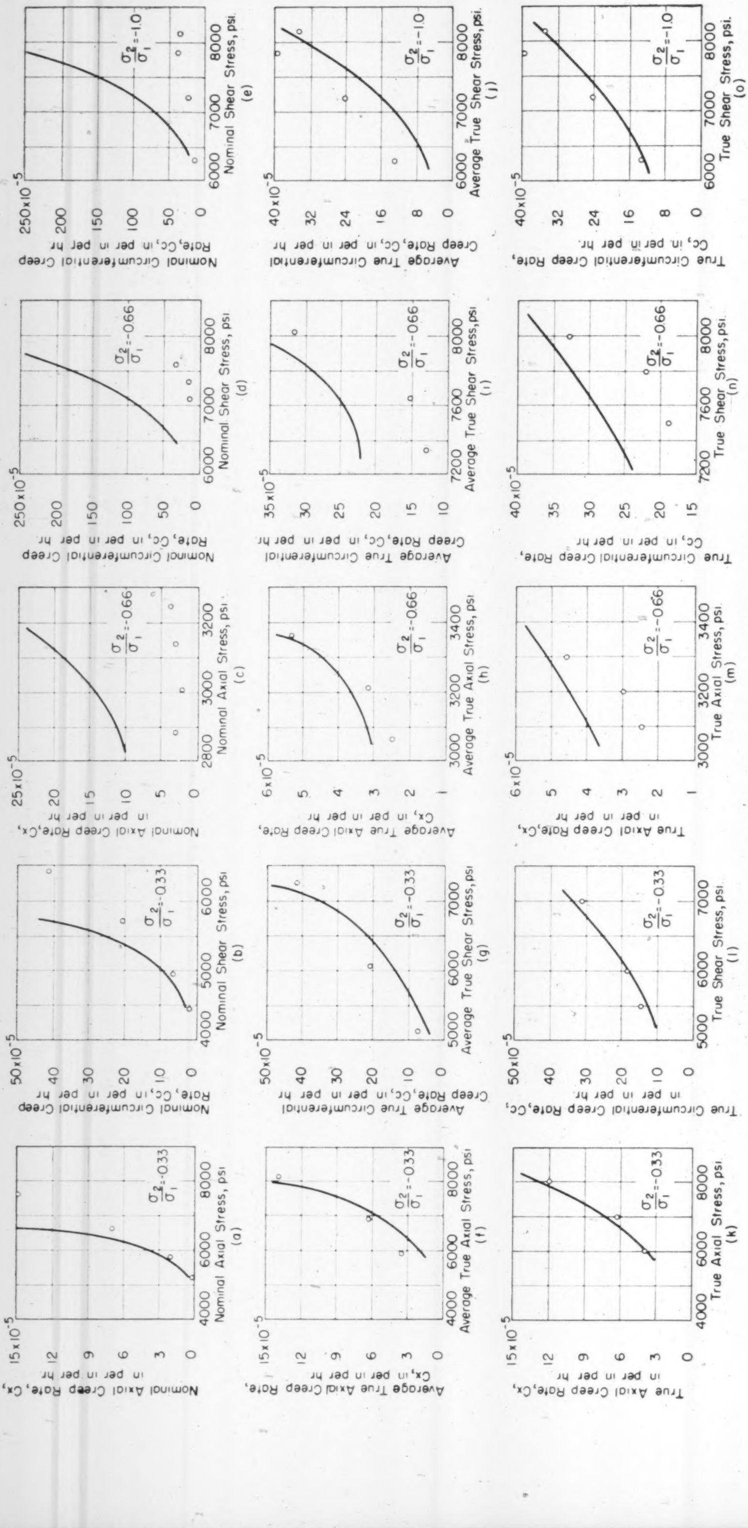


FIG. 17.—Creep Rate-Stress Relations for Combined Stresses.

TABLE III.—COMPARISON OF EXPERIMENTAL AND THEORETICAL CREEP RATES.
(Based on method given in Appendix.)

Nominal Stress Ratio, $R = \sigma_2/\sigma_1$	Average True Axial Stress, σ_x, psi.	Axial Creep Rate, C_x			Average True Shear Stress, psi.	Circumferential Creep Rate, C_c		
		Theoretical, in. per in. per hr.	Experimental, in. per in. per hr.	Difference, per cent		Theoretial, in. per in. per hr.	Experimental, in. per in. per hr.	Difference, per cent
−0.33	5925	1.63×10^{-5}	3.48×10^{-5}	114	5115	4.22×10^{-5}	7.14×10^{-5}	69
	6915	5.3	6.22	17	6040	13.9	20.8	50
	8147	18.55	14.05	−24	7213	49.3	41.6	16
−0.66	3065	3.07	2.46	−20	7348	22.0	13.0	−41
	3213	3.52	3.12	−11	7656	25.15	15.6	−38
	3363	5.86	5.34	−9	8025	41.9	31.7	−24
−1.0	...	...	...	...	6300	5.9	13.09	122
	...	...	...	...	7200	15.7	24.33	55
	...	...	...	...	7850	29.4	3.48	18
	...	...	...	...	8140	38.3	35.0	−9

and circumferential creep rates become

$$C_x = 7.73 \times 10^{-22}(\sigma_x^2 + 3\tau_{xy}^2)^{1.6}(\sigma_x) \quad (18a)$$

$$C_c = 23.19 \times 10^{-22}(\sigma_x^2 + 3\tau_{xy}^2)^{1.6}(\tau_{xy}) \quad (19a)$$

A comparison is given in Table II of the true theoretical creep rates based on Eqs. 18*a* and 19*a* with the experimental values obtained from the true creep-time curves in Figs. 9 and 11 to 15. An examination of the percentage differences between the theoretical and experimental creep rates in Table II shows that the theory gives a good approximation for predicting the creep rates under combined tension and torsion. In fact, the agreement between tests and theory is very good, considering the assumptions made in the theory and the errors involved in the interpolation and calculation of the true creep-time curves based on constant load tests rather than constant stress tests.

The variation between the observed creep stress and creep rate, based on the true stress-strain data, is shown in Fig. 17.

A modification of the foregoing method of interpretation is given in the Appendix.

CONCLUSION

Based upon creep-time data for various ratios of biaxial stresses from 0 to −1.0, and for the aluminum alloy 2S-0 tested, it was found that the Modified St. Venant Theory is a good approximation for predicting creep rates under combined stresses.

Acknowledgments:

The authors express their appreciation to the duPont Co. and to the Research Corp. for their cooperation and for the financial aid that made possible this investigation.

APPENDIX

METHOD OF INTERPRETATION BASED ON "AVERAGE" TRUE STRESS-TRUE STRAIN CURVES

Another method of interpretation of the test data applied in this investigation is based on the determination of true creep-time curves corresponding to the nominal creep-time curves in Figs. 4 to 8. That is, test points corresponding to the test points on the creep-time curves of Figs. 4 to 8 are determined based on Eqs. 15 and 16. The nominal stress on these true creep-time curves is replaced by an average true stress equal to one-half the sum of the original true stress and the final true stress at 100 hr. Values of the creep rate and stress for simple tension are given in Fig. 16 based on the creep-time curves obtained in the foregoing manner. The values of B and n calculated by Eq. 17 were found to be $B = 1.16 \times 10^{-34}$, $n = 7.3$. Using these values of B and n in Eqs. 18 and 19, the theoretical creep rates C_r and C_e were determined for the average true stresses. These theoretical creep rates are given in Table III. The experimental values of the creep rates are also shown in Table III. The percentage difference between the experimental and theoretical values as given in Table III shows that in some cases the discrepancies become large. The variation of the creep rate and average stress is shown in Fig. 17.

Although the foregoing method does not interpolate the test data and does not involve approximations inherent in this interpolation, it appears that the true stress-true creep strain data leads to a more accurate prediction of creep under combined stresses.

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DISCUSSION

MR. H. F. MOORE.[1]—In considering tension-torsion relations it should be remembered that we never find a structural member or a machine part under a purely one-directional *stress*, although it may be under a one-directional *load*. A tension specimen under axial load is subjected to a longitudinal tensile stress, to lateral strains, which seem to indicate internal stresses in the part, although the quantative values of such stresses are not very clearly determined. The specimen also is subjected to diagonal shearing stresses on all planes except those perpendicular or parallel to the direction of the tensile stress. In a torsion specimen there are longitudinal shearing stresses, circumferential shearing stresses, oblique tensile stresses and oblique compressive stresses, and the maximum values of each of these four stresses is the same.

While for many problems of strength of structural or machine parts it is not necessary to consider in detail *all* the stresses in that member, yet we should always keep in mind that the stress pattern is really very elaborate, and, especially in cases of materials which are not equally strong in all directions, we should consider all of the principal stresses set up.

Then too we may well remember that our whole system of computing stresses, strains and strain energies is based on the assumptions: (1) that material is homogeneous, (2) that it is isotropic, and (3) that Hooke's law may be used in computing stress. While none of these assumptions is rigidly correct they have proved very useful as the basis of stress, strain, and strain energy computations which have enabled us to design safe structures with a very high degree of probability of satisfactory service. However with the growing importance of ability to resist repeated stress and to resist creep, increased care is necessary that we examine our structural parts carefully to see whether inhomogeniety, non-isotropy of material and serious deviation from Hooke's law are factors which cannot be neglected.

MR. JOHN M. LESSELLS.[2]—Mr. Marin is to be congratulated in adding some further data to that already available on the strength problem.

It has been well established[3] that in the case of reversed bending combined with static torsion there are certain factors so far unexplained in that a considerable shear stress can be developed before the endurance limit in reversed bending seems to be affected. A further contribution to this phase of the subject has recently been made by Gough.[4]

It seems to me that more data should be made available to explain the above phenomenon before we can hope to understand the additional complexity introduced by the effect of high temperature.

MR. M. J. MANJOINE.[5]—I notice that

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[1] Research Professor of Engineering Materials, Emeritus, University of Illinois, Urbana, Ill.

[2] Associate Professor, Dept. of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[3] J. Marin, "Mechanical Properties of Materials and Design," McGraw-Hill Book Company, Inc., New York, N. Y., p. 167 (1942).

[4] H. J. Gough, "Engineering Steels Under Combined Cyclic and Static Stresses," *Journal of Applied Mechanics*, June, 1950, p. 113.

[5] Research Laboratories, Westinghouse Electric Corp., East Pittsburgh, Pa.

in the picture of the machine the lever arm has three knife edges, not on a straight line; this means, of course, that the load will vary as the weights drop. I wonder how often you have to raise the weights to keep the load constant.

MESSRS. JOSEPH MARIN, J. H. FAUPEL, AND L. W. HU (*authors' closure*).—The authors agree with Messrs. Moore and Lessells in the comments they have made.

Mr. Manjoine observes that the three knife edges of the lever arm used to apply the axial load are not on the same level and that the load on the specimen would vary as the weights drop. The variation in the load on the specimen was prevented by periodic adjustments so that the lever remained horizontal.

THE REVERTING OF HARD-DRAWN COPPER TO SOFT CONDITION UNDER VARIABLE STRESS*

By John N. Kenyon[1]

Synopsis

Fatigue tests carried out on long lengths of hard-drawn copper wire show that "islands" of soft copper may develop at the periphery. This reverting to large crystals was found to occur in some 30 per cent of the heats of copper tested at room temperature and in all heats tested at 70 C.

It is believed that many transmission line failures, attributed to mechanical defect in wire, are a direct result of this softening phenomenon. The finding also brings to mind the century-old theory of crystalline failure and suggests some clarification in definition.

On October 24, 1849, the Institution of Mechanical Engineers met in Birmingham (England) and discussed the problem of fatigue failures in metals. The pros and cons of possible crystalline changes, under variable stress, were considered with understanding and scientific detail. One hundred years later, on October 10, 1949, the mechanism of fatigue was reviewed in a most timely paper by Mr. R. E. Peterson[2] at the A.S.T.M. Pacific Area National Meeting in San Francisco. Mr. Peterson's concluding statement (inferentially referring to the ferrous metals) is believed to be basically correct: "... The 'crystallization theory' of a century ago has been proved unsound by metallographic studies."

A Review of Some Pertinent Fatigue Studies on Copper

In 1933, Rolle and Brace[3] published fatigue data on hard-drawn copper, using the R. R. Moore type of machine and the polished test specimen of reduced diameter. The fatigue strength of 17,000 psi., at 400 million stress reversals, is generally accepted as a very good value for oxygen-free high conductivity (OFHC) hard-drawn copper. The authors obtained some information on electric tough pitch (ETP) copper but the data were never published. It is generally known that the *S-N* fatigue curve for the ETP copper was parallel to and a little below the curve for the OFHC copper. The relative positions of the two graphs are indicated in Fig. 1.

In 1935, Kenyon carried out endurance tests on hard-drawn OFHC and ETP copper wire with the arc machine. Fatigue specimens, up to 30 in. long, were subjected to equal stress-reversal intensity wherein failure could occur at any point within this controlling length. The two fatigue curves did not become asymptotic to the abscissa but trended downward to low values. As in the Rolle-Brace study, the curve for the ETP copper was below and parallel to the curve for the OFHC copper, thus indicating the effect of contained oxide (Fig. 1).

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Fatigue of Materials Laboratory, Princeton, N. J.

[2] R. E. Peterson, "Discussions of a Century Ago Concerning the Nature of Fatigue, and Review of some of the Subsequent Researches concerning the Mechanism of Fatigue," ASTM Bulletin, No. 164, February, 1950, p. 50.

[3] S. Rolle and P. H. Brace, "Some Properties of OFHC Copper," Reprint of a paper and discussion presented at the February Meeting of the Am. Inst. Mining and Metallurgical Engrs. (1933).

In 1940, Kenyon carried out fatigue tests on another set of OFHC and ETP hard-drawn copper wire. After 100 × 10^6 stress-reversals, the *S-N* curves assumed nearly horizontal trends and indicated fatigue strengths in step with the Rolle-Brace values; considering that the wire was die drawn and with the usual run of surface imperfections (Fig. 1).

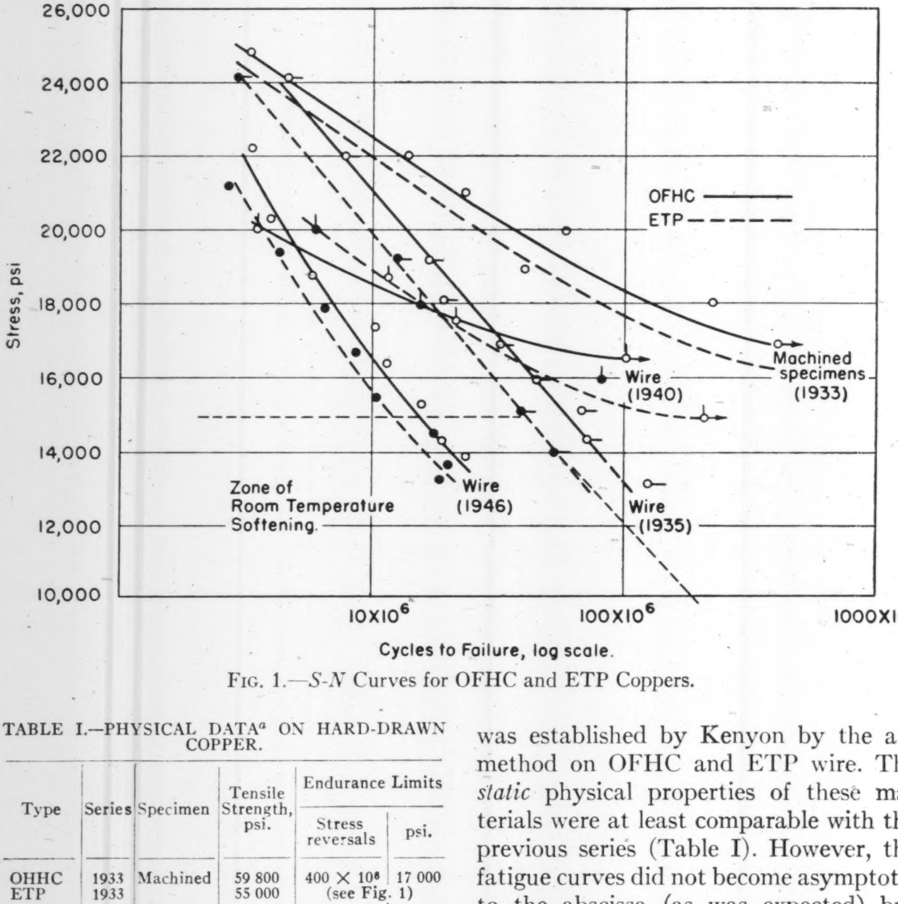

FIG. 1.—*S-N* Curves for OFHC and ETP Coppers.

TABLE I.—PHYSICAL DATA[a] ON HARD-DRAWN COPPER.

Type	Series	Specimen	Tensile Strength, psi.	Endurance Limits	
				Stress reversals	psi.
OHHC	1933	Machined	59 800	400 × 10^6	17 000
ETP	1933		55 000	(see Fig. 1)	
OFHC	1935	Wire[b]	61 000	70	14 700
ETP	1935		59 300	70	13 100
OFHC	1940	Wire[b]	55 900	100	16 700
ETP	1940		65 300	100	15 100
OFHC	1946	Wire[b]	64 500	20	14 300
ETP	1946		64 100	20	13 100

[a] Tested at room conditions.
[b] No. 12 Wire—0.0805 in. diameter.

In 1946, a third set of fatigue curves was established by Kenyon by the arc method on OFHC and ETP wire. The *static* physical properties of these materials were at least comparable with the previous series (Table I). However, the fatigue curves did not become asymptotic to the abscissa (as was expected) but trended downward to abnormally low values. As previously, the relative positions of the two curves indicated the effect of contained oxide. Half-lengths of the broken fatigue specimens were sent to Mr. L. R. Jackson, Battelle Memorial Inst., and the other halves were retained for microscopical studies. The two fatigue curves are shown in Fig. 1.

The microscopic examinations of the fatigued wire tested in 1946, revealed that the hard-drawn copper had undergone a physical change in structure. Small overlapping "islands" of soft material had formed at the periphery, whereas no large crystals were discernible in the stock material. The soft copper had a Knoop hardness of 67 to 75 as compared with a hardness of 132 to 138 for the harder copper.

A subsequent microscopic examination of the broken wire specimens, tested in 1940, did not reveal crystalline alteration; even in the specimens which had been subjected to the highest stress or to over 100 million cycles. Unfortunately, the broken specimens for the series tested in 1933 and 1935 were not retained—they might have given additional microscopical information. It should be noted that the wire series were tested with the arc machine, at room temperature, and at the same speed of rotation.

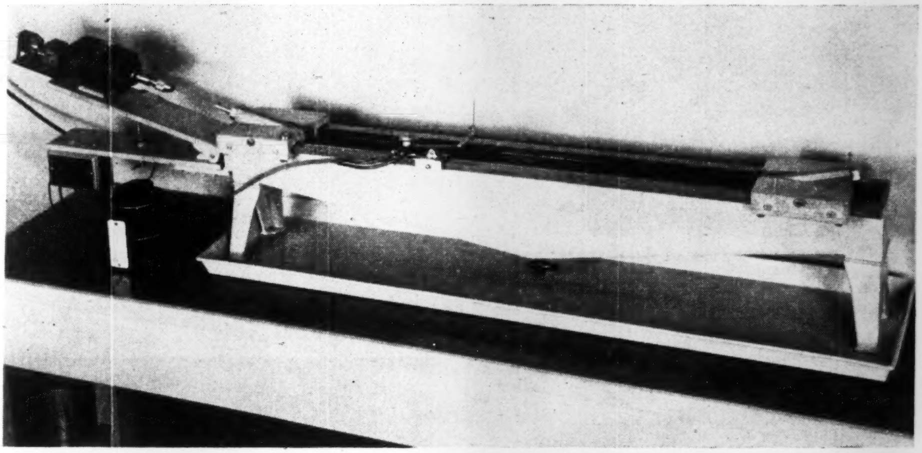

FIG. 2.—Rotating-Wire Arc Fatigue Machine.

THE ROTATING-WIRE ARC FATIGUE MACHINE

The question has been raised (and with very good reason) why other investigators have not reported the formation of large crystals in hard-drawn copper. Other investigators have reported wide divergencies in the fatigue strengths of various heats with similar *static* physical properties. The fact that soft copper formation has not been previously reported may be due to the method of fatigue testing.

The standard machine requires a test specimen wherein the fracture is confined to the center of length. Obviously, the metal on either side of this break is subjected to lower fatigue stress. When a microscopic examination is made, of necessity of the under-fatigued metal, large crystals might not be in evidence. The fatigue machine used in this study (Fig. 2) subjects a long length of wire to equal stress reversal.[4, 5, 6] When large crystals appear in the wire, structural

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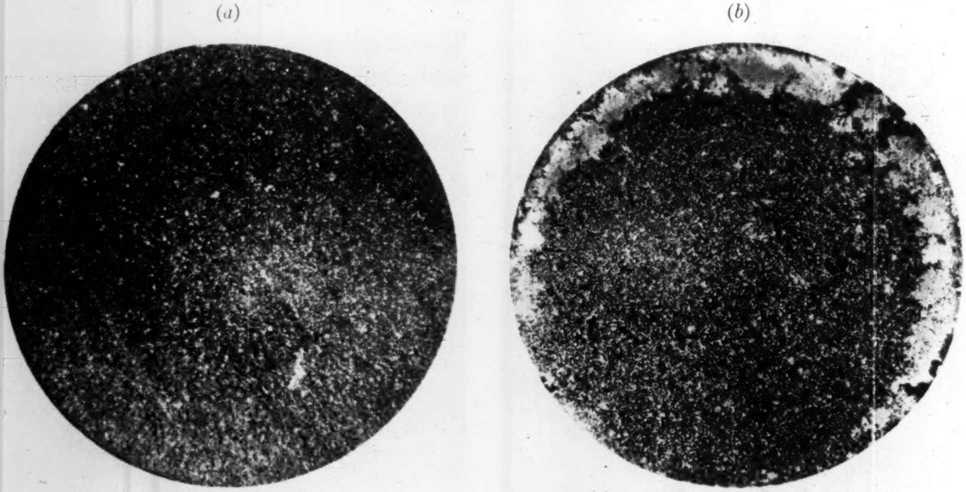
[4] John N. Kenyon, "A Rotating-Wire Arc Fatigue Machine for Testing Small Diameter Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 156 (1935).

[5] John N. Kenyon, "A Corrosion-Fatigue Test to Determine the Protective Qualities of Metallic Platings," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 705 (1940).

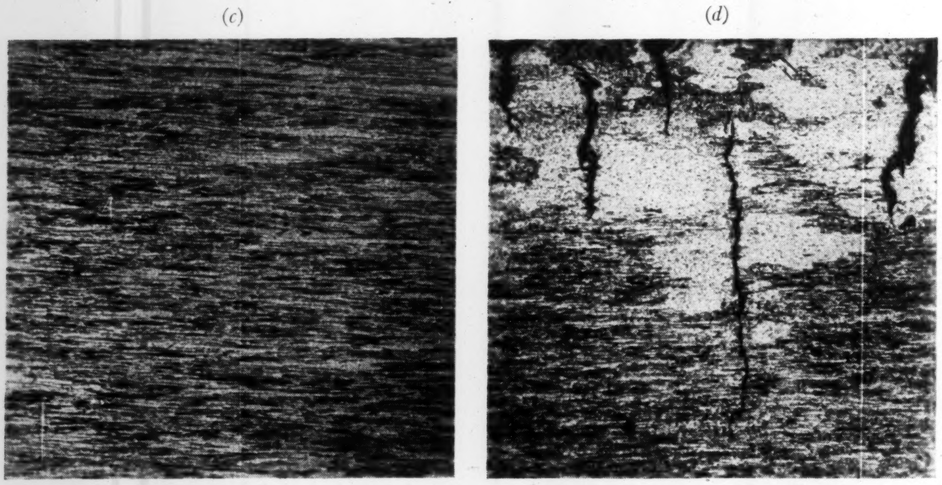
[6] John N. Kenyon, "The Fatigue Properties of Some Cold-Drawn Nickel Alloy Wire," *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 765 (1943).

alteration is in evidence at whatever point the fatigued specimen is microscopically examined.



(a) Cross-section view of stock material.

(b) After 23.9 × 10^6 stress-reversals at 13,900 psi (room conditions).



(c) Longitudinal view of stock material.

(d) After 23.9 × 10^6 stress-reversals at 13,700 psi (room conditions).

FIG. 3.—OFHC Hard-Drawn Copper Wire, 1946 Series (×40).

SOME EVALUATION OF DATA

One outstanding characteristic of the data, shown in Fig. 1, is the four sets of closely paralleling fatigue curves. Each curve for ETP copper appears to be separated from the companion curve for OFHC copper in the order of the *contained oxide*. The four lots of hard-drawn copper materials also show a wide divergence in the fatigue strengths. Since each set was prepared at the same time, in the same mill, and assumedly with the same technique, there is reason to believe that "processing" may be a controlling factor.

The "Zone of Room-Temperature Softening," shown in Fig. 1, is empirically established from the endurance data obtained on some 20 heats of hard-drawn copper wire. Soft copper was discernable in the broken fatigue specimens of every heat of copper whose endurance curve entered this area. This also applies to the higher stressed specimens whose points on the curve are outside, and well above, the empirically established zone.

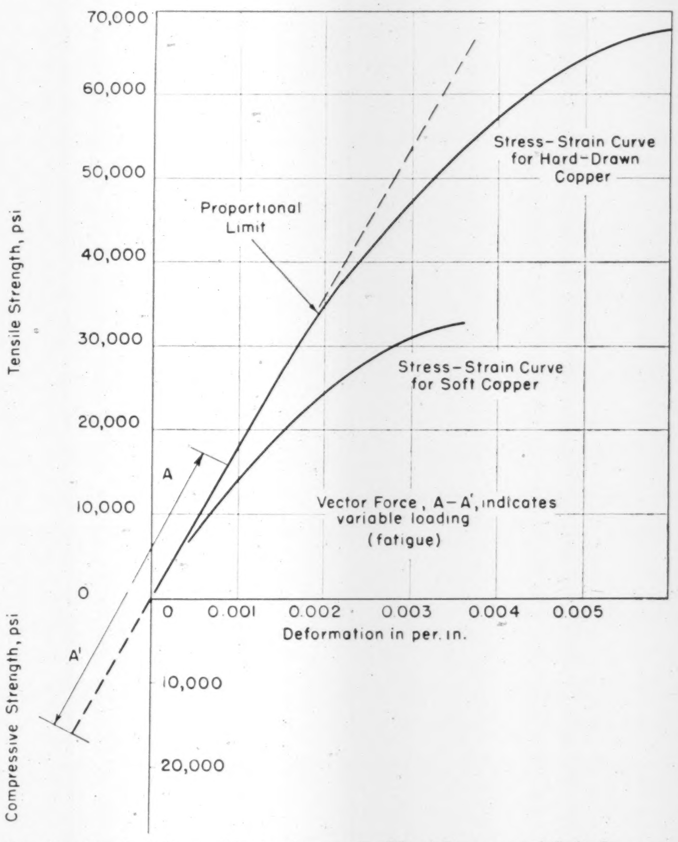


FIG. 4.—Typical Stress-Strain Curves for Hard-Drawn and Soft Copper.

Figure 3 shows the cross-sectional and longitudinal appearance of OFHC copper before and after fatigue testing at room conditions (see 1946 series of Fig. 1). The micrographs of the fatigued material were taken 0.5 in. from the point of fracture after 23.9 million stress reversals at 13,900 psi. Note especially the overlapping "islands" of softened copper at the periphery of the wire (Fig. 3(*b*)) and how the fatigue cracks have propagated into the harder copper (Fig. 3(*d*)).

The mechanism of fatigue failures due to large crystal formation can be clarified by considering the typical stress-strain curves for hard-drawn and soft copper (Fig. 4). A hard copper has definite elastic properties for a given range of stress, while the elastic properties of soft copper are indefinite. The hard copper could be subjected to alternate stress, say A-A', without exceeding the yield strength, but the fatiguing forces, A-A', would have to be considerably reduced to keep within the yield strength of the soft copper. In other words, when a hard-drawn copper is subjected to variable stress and soft crystals develop at the periphery, the fatigue strength is lowered to that of soft copper. Consequently, fatigue cracks (having incipient beginning in soft crystals) propagate into the harder copper and lead to ultimate failure. This propagation is illustrated in Fig. 3(*d*).

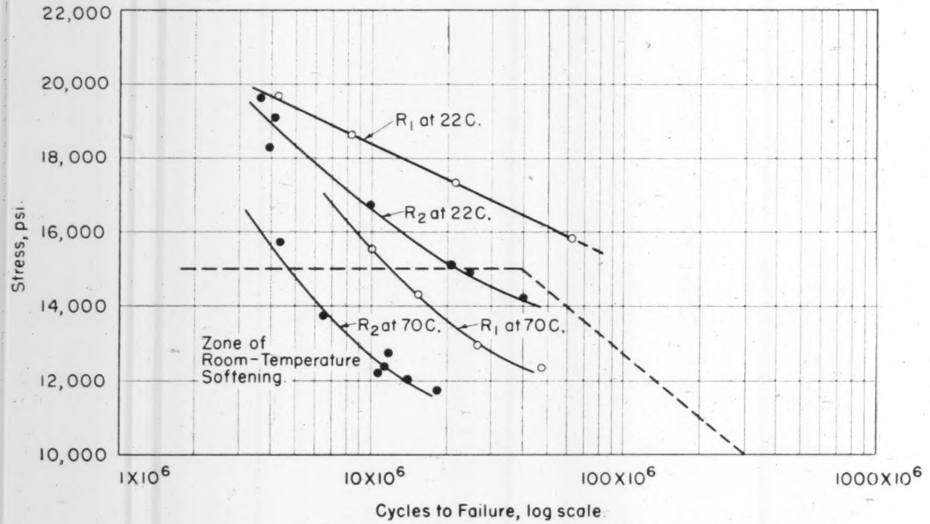


FIG. 5.—*S-N* Curves for Hard-Drawn Copper Wire by the Arc Method.

TABLE II.—PHYSICAL DATA ON "REGULAR" HARD-DRAWN WIRE.

Designation	Specimen	Tested at	Tensile Strength, psi.	Endurance Limits at 13,700 psi.
R_1	Wire[a]	70 C.	68 900	19 × 10^6
R_2	Wire[a]	70 C.	67 700	7

[a] No. 12 wire —0.0805 in. diameter.

LARGE CRYSTAL FORMATION VERSUS LINE SERVICE FAILURES

A British publication[7] of 1944 gives 24-hr. annealing data on several grades of hard-drawn electrical transmission wire. No essential changes in tensile properties were found for temperatures up to 150 C. The conclusion was drawn (in private communication) that the majority of line service failures must be due to mechanical defect in stock material.

In the present study some 175 ft. of copper wire was subjected to the fatigue stressing wherein failure could have occurred at any point within this controlling length. No defect of a mechanical nature was encountered. This leads to the supposition that most line failures are initially due to a fatigue beginning but are listed as defects in stock material because of the *weathered* appearance of the fracture. In fact, most laboratory fatigue fractures give the appearance of original defect due to the discoloration and the "anvil" effect at the point of incipient beginning. Line service failures are believed to follow this cycle of propagation: (*a*) soft crystals of microscopical dimensions develop under cyclical stress because of wind sway and vibration, (*b*) incipient cracks have origin in this low-fatigue-resisting material, (*c*) the cracks propagate into the harder copper thus reducing the cross-section, and (*d*) ultimate failure occurs due to overtension resulting from extreme cold, high wind, or ice accumulation.

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[7] G. W. Preston and H. G. Taylor, "Copper Conductors for Overhead Lines," *Journal*, Inst. Electrical Engrs., Vol. 91, Part II, No. 23, p. 451 (1944).

ENDURANCE TESTS AT HIGHER TEMPERATURE

In order to determine the effect of a moderate temperature increase (70 C.) on the fatigue strength, tests were carried out on two "regular" hard-drawn copper wires which met with the electrical transmission requirements. The data are plotted in Fig. 5 and summarized in Table II.

Three fatigue curves entered the empirically established "Zone of Room-Temperature Softening." One of these curves was established under room conditions and the other two were established at 70 C. As anticipated, the broken specimens showed "islands" of softened copper (Figs. 6 and 7). While the actual *amount* of altered copper was under 5 per cent, it occurred at the periphery and was a controlling factor in a considerably lowered fatigue strength. The fourth fatigue curve, which was established at room conditions, did not enter the empirical "Zone," and no soft copper was in evidence in the tested specimens.

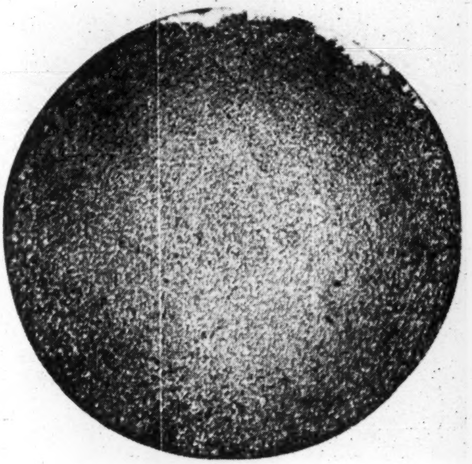

FIG. 6.—Designation R_1 Hard-Drawn Copper Wire After Failure at 15.3 × 10^6 Stress Reversals, 14,300 psi. at 70 C. (×40).

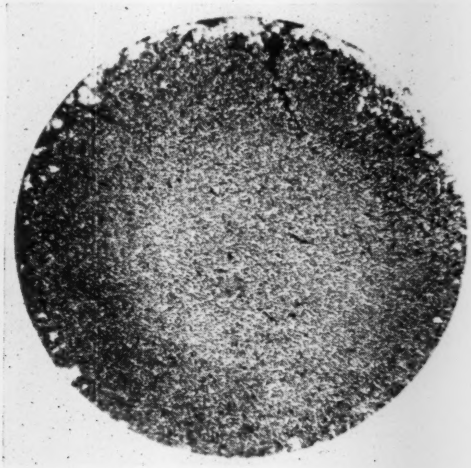

FIG. 7.—Designation R_2 Hard Drawn Copper Wire After Failure at 11.7 × 10^6 Stress Reversals, 12,700 psi. at 70 C. (×40).

SPONTANEOUS SOFTENING OF HARD-DRAWN COPPER

It is generally recognized that certain hard coppers may recrystallize spontaneously at room conditions and eventually revert to soft metal. Two British investigators reported the occurrence in a cold-rolled strip wherein the "... average grain diameter of the crystals had been reduced to about 0.008 mm., or less"[8] (Fig. 8). The finding would seem to indicate that the wire rod with smaller grain diameter might give the lower fatigue strength. The opposite effect was encountered in the present study.

The two "regular" coppers had rod

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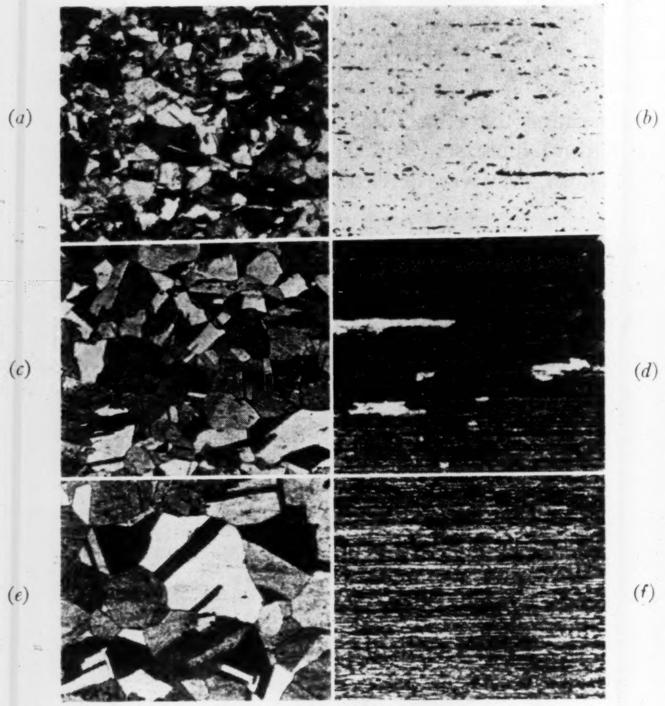
[8] M. Cook and T. L. Richards, "The Self-Annealing of Copper," *Journal*, Inst. Metals, Vol. 73, p. 1 (1943).

grain diameters of about 0.015 mm. and 0.025 mm., respectively. Both were drawn to No. 12 wire in the same mill and with the same number of passes. The hard-drawn copper wire, from the rod with grain diameter of 0.015 mm., had a considerably higher fatigue strength. (Figs. 5, 9 and 10).



(*a*) Before rolling.
(*b*) After rolling. Completely reverting to soft copper in 15 months

(*c*) Before rolling.
(*d*) After rolling. Partial reverting to soft copper in 15 months

(*e*) Before rolling.
(*f*) After rolling. No reverting to soft copper in 15 months

Fig. 8.—Showing How a Hard-Drawn Copper Strip Spontaneously Reverts to Soft Copper at Room Conditions When the Grain Size is Below a Certain Critical Value.

Clarification in Definition of "Crystalline" Failure

The century-old theory of "crystalline" failure (according to the concensus of opinion) is the hardening of a metal under cyclical stress, a loss in ductility, assumption of cast-iron-like properties and a general deterioration in the fatigue strength. Such a theory of service failure has been proved to be unsound.[2] On the other hand, it is felt that some terminology by way of definition should be considered for fatigue failures in certain non-ferrous metals (such as lead and copper) wherein there have been structural changes. Should the mechanism of failure be termed one of crystallization, recrystallization, or crystalline re-orientation. The question is propounded and is believed worthy of discussion in open forum.

SOME GENERAL CONCLUSIONS

A hard-drawn copper is in metastable condition and the British finding has demonstrated that spontaneous softening can occur under static room conditions. Inferentially, the softening tendency might be accelerated under vibrational stress and in the nearly complete absence of the so-called "annealing" temperature.

The four sets of paralleling curves (Fig. 1) indicate that processing may be an important factor in arriving at the mechanism of large crystal formation. A consideration of the contained impurities (or added elements, depending on the point of view) must be reserved for a more intensive study. For further work, the following leads appear to be worthy of careful consideration: (*a*) pulsating-tension fatigue tests to determine whether the formation of "islands" of soft copper is a surface effect, (*b*) effect of contained impurities, (*c*) grain diameter as suggested by the British study, (*d*) effect of processing as suggested by the parallel curves in Fig. 1, and (*e*) nature of the solid solutions of the contained elements.

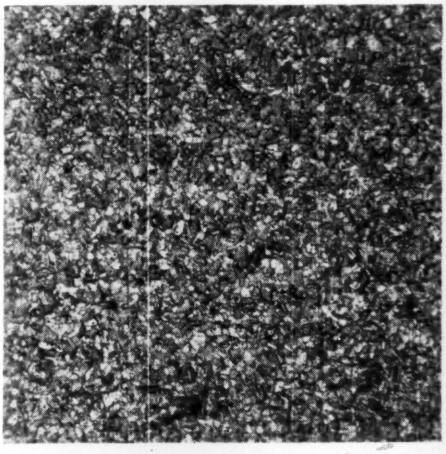


FIG. 9.—Designation R_1 Copper, $\frac{5}{16}$-in. Rod Before Rolling, With Average Grain Diameter of 0.015 mm.

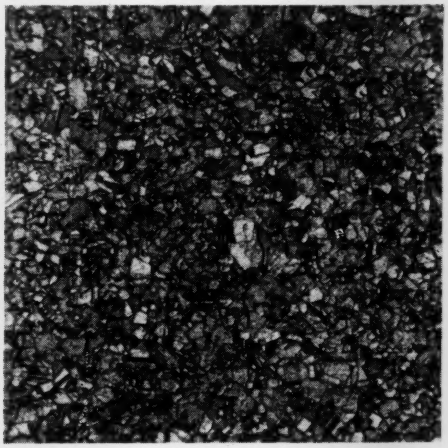


FIG. 10.—Designation R_2 Copper, $\frac{5}{16}$-in. Rod Before Rolling, With Average Grain Diameter of 0.025 mm.

Acknowledgment:

Appreciation is expressed to H. F. Moore for constructive suggestions on the preparation of the paper, and to R. M. Sample, Sample-Marshall Laboratories, for suggestions and the contribution of the micrographs.

DISCUSSION

Mr. J. S. Smart.[1]—Mr. Kenyon has presented evidence of the occurrence of partial recrystallization in certain copper wires being tested in fatigue within the normal operating temperature range for line wire. The fact that all lots tested did not partially recrystallize is significant, and while variations in grain size have been noted, it seems more probable to ascribe the major cause to differences in the annealing characteristics of the various lots of wire under test. If this proves to be the case, the use of coppers having high recrystallization temperatures, of which there are a number of types, may be indicated where resistance to fatigue is important.

In order to obtain more evidence on the importance of annealing temperature, which depends on impurity content and thermal history, we have furnished Mr. Kenyon with a quantity of high-purity copper wire having a very much lower recrystallization temperature than the previous coppers tested. These test results should be of considerable interest when they become available.

Mr. J. N. Kenyon (*author*).—The following table gives some fatigue values obtained on the spectrographically pure hard-drawn copper wire submitted by Mr. Smart: The loss in fatigue strength with temperature rise to 70 C. is considerably greater than was obtained on commercial line wire. This indicates that at least one so-called impurity must be a valuable addition agent.

Strength of Stock, psi.	Tested at	Fatigue Stress, psi.	Cycles to Failure	Loss, per cent
56 400	22 C.	14 700	17.2×10^6	
	70 C.	14 700	1.7	90.0
	22 C.	14 300	23.0	
	70 C.	14 300	3.5	85.0
				Avg. 88.0

Mr. G. R. Gohn.[2]—I should like to add to what Mr. Smart has just stated the fact that one constituent of copper which we do not normally consider an impurity, namely, silver, is frequently reported as copper and has a most marked effect on the annealing characteristics. It is rather unfortunate that the compositions of these various coppers were not reported. That might be the clue to what is happening.

Mr. Kenyon (*author*).—Tests carried out on "Lake" copper (relatively high in silver) indicate average low fatigue strengths. The values are very much better than those obtained on high-purity copper. The chemical contents and processing techniques are not reported because some were not available. These data must be carefully considered in future fatigue studies.

Mr. John M. Lessells.[3]—The paper by Mr. Kenyon is of considerable interest, discussing as it does the effect of impurities.

It is interesting to note that Gough[4] has shown that copper has its elastic properties changed by the effects of cyclic stress. In the annealed state there was no proportional limit observed in the static tension test whereas after cyclic stress the material did show a definite proportionality limit.

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[1] Research Dept., American Smelting and Refining Co., Barber, N. J

[2] Member of Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[3] Associate Professor, Department of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[4] H. J. Gough, "Elastic Limits of Copper Under Cyclical Stress Variation," *Engineering*, September 8, 1932.

Comparing the fatigue results of annealed and cold-drawn copper, it was shown that for the annealed material the knee of the *S-N* diagram occurred before 20 × 10^6 cycles but there was no knee on the curve for the cold-drawn material at 500 × 10^6 cycles.

Whether these results can be applied to explain the effects of impurities as found by Mr. Kenyon remains to be seen. They do indicate that the elastic and fatigue characteristics of copper can be radically changed by the effects of cold work produced either by cyclic stress or by cold drawing.

MR. KENYON (*author*).—Gough's finding that soft annealed copper develops a definite proportional limit under cyclic stress, would seem to suggest a cold-working effect. The fact that soft copper makes appearance in a hard-drawn material under cyclic stress would seem to indicate some extreme in metastable condition. This condition may be due to an over-drawing, an over amount of some impurity, or impurities not in solid solution.

MESSRS. G. M. SINCLAIR AND T. J. DOLAN[5] (*by letter*).—The present study by Mr. Kenyon on the recrystallization of copper wire subjected to fatigue stressing is very important and deserves the attention of all who are interested in the problem of the fundamental mechanism of fatigue failure.

The findings of this paper raise a question somewhat akin to the ancient one, "Which came first, the chicken or the egg?" In the present situation it would be instructive to know whether the large recrystallized grains or the fatigue crack appear first during the process of fatigue failure. In the Edgar Marburg Lecture before the American Society for Testing Materials in 1933,[6] H. J. Gough summarized a number of experiments from whith it appeared that fatigue failure is usually preceded by slip and crystal breakdown (cold working) of the metal. From this, one might expect that the occurrence of microscopically visible recrystallization *during* a fatigue test is unlikely since any such structure should be rapidly broken down by the succeeding cycles of reversed stress.

The occurrence of recrystallization *after* the failure has taken place is also a possibility and should be considered. In 1948, a limited study of the fatigue recrystallization of commercial grade, tough-pitch copper wire was carried out in the Talbot Laboratory of the University of Illinois. Wire specimens in the half-hard-drawn condition were tested in a rotating-arc fatigue machine similar to that used by Kenyon. Metallographic examination of the wire after repeated stressing showed recrystallized zones adjacent to the fractured ends of several specimens which were broken at relatively high stresses. No microscopic evidence of recrystallization was apparent in those specimens which were interrupted before fracture had taken place. In those cases where recrystallized zones were found in the microstructure after fracture, it was noted that a sharp increase in temperature of the specimen occurred just *prior* to failure. This was presumed to be due to the severe working received by the reduced cross-section of the metal as the fracture developed. The temperature of the fractured ends of these specimens immediately after failure was found to be well over that necessary to recrystallize the parent material.

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[5] Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[6] H. J. Gough, "Crystalline Structure in Relation to Failure of Metals—Especially by Fatigue," *Proceedings*, Am. Soc. Testing Mats., Vol. 33, Part II, p. 3 (1933).

Mr. Kenyon's comments regarding the points in question will be greatly appreciated.

MR. KENYON (*author*).—Traces of soft copper can usually be detected in the commercial grades of hard-drawn wire. These microscopic areas were no more in evidence in the material that reverted to soft copper under cyclic stress than in the heats of copper which did not revert. Some recrystallization may take place after fatigue failure. However, when soft copper did appear, the phenomenon was always associated with an abnormally low fatigue strength.

MR. H. F. MOORE[7] (*by letter*).—The presence of large grains of relatively soft copper is a very interesting phenomenon, and Mr. Kenyon is to be thanked for bringing it to the notice of the Society.

In Mr. Kenyon's paper it was stated that in the stock metal before testing no such large grains were found. Some further details of the search for such large grains in unstressed metal would be of interest.

The question arises whether such large grains may not have been formed by the high localized temperatures set up in the copper as it was subjected to repeated stressing in the plastic range of the metal. However, the discussion of Mr. Kenyon's paper by Messrs. Sinclair and Dolan treats of this matter of localized high temperature, and it is not further discussed here except to point out that Mr. Kenyon's testing machine was run at 3600 rpm., which would encourage high-temperature development of large soft grains of copper. Possibly both localized high temperature and cycles of stress are effective.

Mr. Kenyon states that for the hard copper the "proportional limit" was of the order of 32,000 psi., but that the soft copper showed no definite "proportional limit," and that accordingly its endurance limit must be indefinite and below 10,000 psi. The writer of this discussion doubts the significance of the proportional limit" as an index of fatigue strength. In fact, the term "proportional limit," used without any statement of sensitivity of apparatus used in testing for it, or of any definite deviation from Hooke's law used to locate it, seems to the writer to be of doubtful quantitative value for predicting any kind of structural strength of a material.[8]

MR. KENYON (*author*).—The stress-strain diagrams exemplify how the physical properties (including fatigue strength) fall off when a hard copper reverts to soft material. Since the soft crystals appear at the periphery, they are a controlling factor in the fatigue strength of the hard-drawn wire.

The British study demonstrates that a hard copper may completely revert to soft material under static room-temperature storage. It is but logical to assume that the phenomenon might be considerably accelerated by cyclic stress and in the nearly complete absence of the so-called annealing temperature.

(*author's closure*).—The several discussions are most suggestive in leads for future studies. Of immediate interest is the employment of a pulsating-tension machine wherein a large percentage of the fatigue breaks occur clear of the grippings. In that this method subjects the entire cross-section of the wire to the desired fatigue stress, important information should be obtained on whether the "islands" of soft copper are essentially a surface phenomenon.

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[7] Research Professor of Engineering Materials, Emeritus, University of Illinois, Urbana, Ill.

[8] Cyril Stanley Smith, "Fatigue Tests of Some Copper Alloys," *Proceedings*, Am. Soc. Testing Mats., Vol 41, p. 849 (1941).

THE FORMING CHARACTERISTICS OF BERYLLIUM COPPER STRIP*

By J. T. Richards[1] and E. M. Smith[1]

Synopsis

The results of tests to correlate the forming characteristics of beryllium copper strip prior to precipitation hardening with usual specification properties including tensile strength, elongation, and Erichsen cup test are reported. Formability is expressed as the minimum safe radius for forming a 90-deg. bend by means of a punch and die set-up. The effects of temper, stock thickness, grain size, and grain direction upon formability and miscellaneous design or specification properties are noted.

The minimum forming radius varies directly with stock thickness; however, no simple correlation was found relating formability with usual acceptance tests. The best combination of formability and tensile properties was obtained from material having a relatively fine grain structure. Although directionality is apparently absent in solution treated and quarter hard strip, it may be a factor in harder tempers.

Precipitation-hardening beryllium copper offers certain advantages in the production of a wide variety of flat springs, clips, contacts, diaphragms, and small structural members, since it can be severely formed in the unhardened condition and then heat treated to give a high degree of strength and hardness. Parts are generally produced from strip by blanking, punching, bending, and drawing. Consequently, an indication of the forming characteristics of beryllium copper strip is important from design, material selection and manufacturing considerations.

Although strip may be cupped, swaged, dished, crimped, corrugated, or drawn during fabrication, experience indicates that bending properties are the limiting factor in the greatest number of applications. Therefore, an evaluation of the bending characteristics provides an appropriate starting point in a study of formability.

There are available both direct and indirect methods for determining safe forming radii for various materials in strip form. In the direct method (**1, 2, 3**),[2] strip specimens are formed to a 90-deg. bend by means of a punch-and-die setup. This test procedure is not only rapid but also provides a direct measure of bending characteristics.

Most of the indirect methods attempt to correlate the results of tension tests with formability. Tensile elongation, reduction of area, and the yield strength–tensile strength ratio have been suggested as indices for predicting relative bending properties. Failure to determine a simple correlation probably stems from the many variables associated with shop forming operations and conventional tension testing.

In considering the tension test, the reduction of area (sometimes referred to as zero-gage-length elongation, see pages 54 and 55 of paper by Low and Prater (**6**)) provides an approximate measure of the ability of a material to withstand plastic deformation. The yield-tensile ratio offers similar information, since a low yield strength in combination with a high tensile strength may indicate considerable capacity for cold work.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Development Engineer, and Metallurgist, respectively, The Beryllium Corp., Reading, Pa.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1099.

Tensile elongation cannot serve as a measure of formability unless certain allowances are made. Elongation values are greatly influenced by variations in grain size, grain direction, and specimen dimensions; and, in addition, past work indicates that different alloys do not respond in the same manner to these variables. For example, Walker and Craig (**4**) have reported an increase in elongation with an increase in grain size for brass, while Brewer (**5**) found a drop in elongation with an increase in grain size for an aluminum alloy. The effect of grain direction also varies, since Low and Prater (**6**) obtained higher elongation in longitudinal specimens of an aluminum alloy, whereas Gohn and Arnold (**7**) found that for several copper alloys transverse tests gave the highest elongation. There is general agreement, however, that increases in cross-sectional area resulting from added thickness or width give higher elongation values (**8, 9, 10, 11**).

The effect of gage length upon elongation is of particular significance, since the effective gage length in forming strip is generally much less than the standard 2-in. gage length usually employed in tension testing. Several investigators (**9, 11, 12**) have shown that elongation over 2 in. does not serve as an indication of the elongation over other gage lengths, particularly short ones. Several methods (**13, 14**) have been devised for predicting minimum forming radii, based on elongation over short gage lengths.

In the present investigation, it was decided to concentrate on the direct method employing a punch-and-die setup, since this procedure approximates actual fabricating practice. At the same time, the direct test offers a simple means of measuring springback, an important factor in all forming operations. Tension tests were also conducted as a means of verifying the indirect methods as applied to beryllium copper. In addition, Erichsen cup tests were also included, since this test may be incorporated in specifications to insure a certain degree of formability.

To obtain a complete picture, the influence of temper, strip thickness, grain size, and grain direction upon bending properties was investigated. The effect of width was not considered, because most beryllium copper parts are produced from strip rather than sheet. Since beryllium copper is generally fabricated prior to heat treating, tests were not conducted in the precipitation-hardened condition.

MATERIALS

The alloy studied in this investigation was the standard 2 per cent beryllium copper material meeting the requirements of A.S.T.M. Specification B 194 – 49 T.[3] Ninety-eight different lots of strip, representing various tempers, grain sizes, and thicknesses, were tested prior to precipitation hardening. Grain sizes varied from 0.010 to 0.120 mm., while strip thicknesses ranged from 0.005 to 0.042 in.

Lots having grain sizes from 0.025 to 0.045 mm., inclusive, were taken from stock and can be considered typical of commercial material. Other lots were specially prepared to give the desired range of grain sizes. Although chemical analyses were not undertaken at the time of test, the composition of all lots was previously determined at the ingot stage and fell within the limits listed in Table I.

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[3] Tentative Specification for Beryllium-Copper Alloy Plate, Sheet, and Strip (B 194 – 49 T), 1949 Book of A.S.T.M. Standards, Part 2, p. 267.

TEST METHODS

Three methods of test were employed: tension, Erichsen, and the simple 90-deg. cold-bend test. Average grain sizes were estimated by the comparison method outlined in A.S.T.M. Method E 79 – 49 T.[4] Since the Erichsen test is well known, further discussion of this method is not warranted.

The tensile strength was determined in accordance with the Standard Methods of Tension Testing of Metallic Materials (E 8 – 46),[5] using cross-milled specimens similar to Fig. 2 of that method with length reduced to 6 in. to permit removal of transverse specimens from 6-in. wide strip. Tension tests were conducted on a 5000-lb. Olsen testing machine. The proportional limit and various yield strength values were taken from stress-strain curves drawn with an Olsen automatic recorder. Several check runs were made with an Olsen dial extensometer, and good agreement was found between this method and the recorder.

TABLE I.—CHEMICAL COMPOSITION.

Beryllium, per cent	1.90 to 1.99
Cobalt, per cent	0.23 to 0.31
Iron, per cent	0.07 to 0.12
Silicon, per cent	0.07 to 0.13
Aluminum, per cent	0.05 to 0.08
Nickel, per cent	0.00 to 0.02
Tin, per cent	trace
Lead, per cent	0.00
Zinc, per cent	0.00
Copper, per cent	remainder

Since the value of the proportional limit is dependent upon the sensitivity of the test method, it is qualified herein as the stress at 0.002 per cent offset. This offset represents approximately the width of the stress-strain curve drawn by the recorder, and the proportional limit is calculated from the load at the point where the curve leaves the modulus line.

In determining reduction of area, the original thickness, original width, and final thickness were measured by micrometer, while the final width was determined microscopically. The value obtained is, at best, an estimate because of the difficulty in accurately obtaining the minimum final cross-sectional area.

The elongation in 0.2 in. was also measured microscopically. Lines perpendicular to the longitudinal axis of the specimen were scribed 0.2 in. apart over a 2-in. gage length. Determinations were made over the 0.2-in. gage length containing the fracture, while the elongation in 2 in. was taken on the same specimen. To determine elongations over other gage lengths, several specimens were scribed with lines 0.1 in. apart, and special tests were also conducted on similar material with gage lengths up to 10 in.

The 90-deg. cold-bend test utilizing a punch-and-die set-up has been described in detail elsewhere (**1**, **2**, **3**). Sheared specimens, $\frac{1}{2}$ by 2 in., were removed from test strips at 0, 45, and 90 deg. to the direction of rolling. Unclamped specimens were then bent by a sharp, 90-deg. V-groove die and a series of 90-deg. punches having sharp, $\frac{1}{64}$, $\frac{1}{32}$, $\frac{3}{64}$, $\frac{1}{16}$, $\frac{3}{32}$, $\frac{1}{8}$, $\frac{5}{32}$, $\frac{3}{16}$, $\frac{7}{32}$, and $\frac{1}{4}$-in. radii. Since only relatively light gage stock was tested, a hand-operated punch was employed. To determine the effect of punch speed, a series of tests was made; however, no variations in formability or springback were encountered over a fairly wide speed range.

In this study, two end points were used to evaluate forming characteristics: (1) the minimum punch radius without cracking and (2) the minimum punch radius without an "orange peel" effect. A specimen of reduced formability will crack when formed over too sharp a radius. This tendency decreases as the punch radius is increased, until only orange peel can be observed. Further in-

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[4] Tentative Methods for Estimating the Average Grain Size of Wrought Copper and Copper-Base Alloys (E 79 – 49 T), 1949 Book of A.S.T.M. Standards, Part 2, p. 1086.

[5] 1949 Book of A.S.T.M. Standards, Part 2, p. 993.

TABLE II.--TEST RESULTS.

Nominal Thickness, in.	Grain Size, mm.	Angle with Rolling Direction, deg.	Minimum Punch Radius, in.[a]		Springback, deg.	Tensile Strength, psi.	Proportional Limit, 0.002 per cent Offset, psi.	Yield Strength, psi.			Reduction of Area, per cent	Elongation, per cent		Erichsen Value, mm.	Ratio, Yield Strength to Tensile Strength
			Without Cracking	Without Orange Peel				at 0.01 per cent Offset	at 0.2 per cent Offset	at 0.5 per cent Strain		in 0.2 in.	in 2.0 in.		
SOLUTION-TREATED STRIP (CONDITION A)															
0.005	0.030	0	S	S	−10	61 200	...	...	...	...	...	52	41	10.61	...
0.008	0.035	0	S	S	−9	60 300	...	...	...	...	...	73	54	11.80	...
0.010	0.030	0	S	S	−7	64 200	...	...	...	...	...	75	52	10.48	...
0.013	0.035	0	S	S	−7	62 600	...	...	...	...	...	81	55	11.33	...
0.016	0.025	0	S	S	−7	66 200					...	81	52	11.33	...
0.020	0.035	0	S	S	−8	64 500	18 500	23 100	30 400	31 500	80	89	56	12.14	0.36
0.025	0.010	0	S	S	−7	69 100	26 600	29 400	35 100	37 000	82	84	48	12.43	0.43
...	0.025	0	S	S	−7	65 500	17 900	22 000	31 100	32 900	87	88	53	12.51	0.34
...	0.045	0	S	S	−8	63 200	19 700	23 200	31 200	32 600	86	86	55	12.76	0.37
...	0.120	0	S	S	−5	55 600	15 200	19 200	28 600	30 200	90	86	57	12.66	0.35
0.030	0.040	0	S	S	−4	62 200	16 400	19 800	29 600	31 300	93	90	56	12.65	0.32
0.040	0.040	0	S	S	−7	63 900	17 900	21 700	30 000	31 500	95	96	56	12.99	0.34
QUARTER HARD STRIP (CONDITION ¼ H)															
0.005	0.030	0	S	S	−5	74 400	...	...	...	...	...	26	16	5.95	...
0.008	0.025	0	S	S	−8	78 700	...	...	...	...	...	50	26	6.65	...
0.010	0.025	0	S	S	−8	79 300	...	...	...	...	...	57	27	7.38	...
0.013	0.025	0	S	S	−6	77 700	...	...	...	...	...	53	20	6.88	...
0.016	0.035	0	S	S	−7	83 100	52 700	62 800	78 400	78 100	45	55	17	6.74	0.76
0.020	0.035	0	S	S	−6	83 500	56 900	65 700	79 100	78 800	45	66	18	7.35	0.79
0.023	0.010	0	S	S	−6	82 700	59 500	64 900	70 600	70 600	53	56	25	9.18	0.78
...	0.025	0	S	S	−5	79 200	54 100	61 300	71 900	71 700	53	59	21	8.02	0.77
...	0.045	0	S	S	−4	75 300	53 900	59 700	66 700	66 900	67	71	30	8.46	0.79
...	0.120	0	S	S	−6	78 600	53 400	59 200	68 600	68 000	38	57	19	8.75	0.75
0.025	0.025	0	S	S	−4	83 800	58 100	66 700	78 300	77 700	51	56	20	7.86	0.80
0.032	0.030	0	S	S	−7	76 800	45 900	52 100	66 900	67 900	62	72	30	8.98	0.68
0.036	0.025	0	S	S	1	76 600	41 800	47 900	64 500	63 400	64	65	27	9.96	0.63
0.042	0.025	0	S	S	3	73 700	41 600	48 200	63 600	62 800	80	83	28	9.70	0.65
HALF HARD STRIP (CONDITION ½ H)															
0.005	0.035	0	S	S	−7	77 600	...	...	...	...	...	25	9	5.19	...
		45	S	S	−5										
		90	S	S	−4	82 000	...	...	...	...	...	32	10		...
0.008	0.035	0	S	1/64	1	80 300	...	...			...	34	13	5.33	...
		45	S	1/64	2										
		90	S	1/64	3	79 600	...	...	...	.	...	45	15		...
0.010	0.035	0	S	1/64	1	88 100	...	...	...	...	...	36	13	5.44	...
		45	S	1/64	2										
		90	S	1/32	5	93 100	...	...	...	...	...	41	11		...
0.015	0.030	0	S	1/32	2	88 500	...	...	...	...	...	42	14	6.10	...
		45	S	1/32	2										
		90	S	3/64	3	89 500	...	...	..	...	...	47	16		...
0.020	0.015	0	S	1/32	0	79 600	54 200	62 500	72 300	72 100	60	60	24	7.76	0.78
		45	S	1/32	−1										
		90	S	1/32	2										
...	0.025	0	S	3/64	6	90 100	68 400	76 100	88 300	83 700	43	48	12	5.99	0.84
		45	S	3/64	6										
		90	S	3/64	6	92 100	59 400	68 900	87 100	87 100	42	51	11		0.75
...	0.035	0	S	3/64	5	82 000	68 900	74 100	81 300	81 600	39	46	15	7.12	0.90
		45	S	3/64	5										
		90	S	3/64	5	86 800	50 200	60 600	84 400	81 500	50	50	14		0.70
...	0.045	0	S	1/16	8	81 300	56 100	61 700	76 500	75 600	45	50	20	7.56	0.76
		45	S	3/32	8										
		90	S	3/32	9										

TABLE II.—TEST RESULTS.—*Continued.*

Nominal Thickness, in.	Grain Size, mm.	Angle with Rolling Direction, deg.	Minimum Punch Radius, in.[a]		Springback, deg.	Tensile Strength, psi.	Proportional Limit, 0.002 per cent Offset, psi.	Yield Strength, psi.			Reduction of Area, per cent	Elongation, per cent		Erichsen Value, mm.	Ratio, Yield Strength to Tensile Strength
			Without Cracking	Without Orange Peel				at 0.01 per cent Offset	at 0.2 per cent Offset	at 0.5 per cent Strain		in 0.2 in.	in 2.0 in.		
						HALF HARD STRIP (CONDITION ½ H)—*Continued*									
. . .	0.120	0	S	1/16	4	75 500	60 000	65 300	70 600	70 500	31	28	15	6.39	0.88
		45	S	1/16	4										
		90	S	1/16	5										
0.025	0.030	0	S	1/16	6	89 400	58 100	66 800	87 200	84 000	39	46	9	6.88	0.75
		45	S	3/32	7										
		90	S	3/32	12	95 000	62 700	72 400	93 900	88 500	39	56	10		0.76
0.030	0.025	0		3/32	7	83 500	50 500	60 100	79 600	76 000	39	60	19	7.86	0.72
		45	S	3/32	8										
		90	S	1/8	9										
. . .	0.045	0	S	1/8	10	90 600	61 900	70 300	88 900	85 400	38	41	12	6.46	0.78
		45	S	1/8	12										
		90	S	1/8	14	96 000	59 100	67 500	93 900	98 500	44	52	12		0.70
0.036	0.025	0	S	1/8	8	91 600	56 400	65 900	84 900	82 800	41	55	15	7.64	0.72
		45	S	1/8	8										
		90	S	1/8	9	94 600	54 800	65 700	93 100	83 700	40	49	12		0.69
. . .	0.045	0	S	1/8	7	83 200	46 800	54 000	77 700	75 200	53	67	20	8.29	0.65
		45	S	1/8	8										
		90	S	1/8	10										
0.042	0.025	0	S	1/8	2	92 000	54 200	64 400	89 400	78 400	44	54	13	8.15	0.70
		45	S	1/8	7										
		90	S	5/32	8										
. . .	0.045	0	S	1/8	4	82 900	51 700	61 900	80 800	75 900	58	66	20	8.04	0.75
		45	S	1/8	5										
		90	S	5/32	9	85 400	48 900	57 100	81 800	76 300	58	55	17		0.67
						HARD STRIP (CONDITION H)									
0.005	0.025	0	S	1/64	13	110 000	. . .	. . .	. . .	. . .	. . .	15	1.5	3.72	. . .
		45	S	1/32	. . .										
		90	1/64	1/32	. . .										
0.008	0.030	0	S	1/64	6	106 800	. . .	. . .	. . .	. . .	. . .	8	2	4.03	. . .
		45	S	1/32	11										
		90	1/64	3/64	22	112 400	. . .	. . .	. . .	. . .	. . .	18	5		. . .
0.010	0.025	0	S	1/32	7	110 400	. . .	. . .	. . .	. . .	. . .	14	4	4.58	. . .
		45	S	3/64	13										
		90	1/32	1/16	17										
0.012	0.030	0	S	1/32	4	114 700	. . .	. . .	. . .	. . .	. . .	14	2	4.19	. . .
		45	1/64	1/16	14										
		90	3/32	3/32	26	122 900	. . .	. . .	. . .	. . .	. . .	22	3		. . .
0.016	0.015	0	S	1/16	8	111 100	88 800	99 500	110 700	102 400	19	32	4	5.41	0.90
		45	S	3/32	14										
		90	1/32	3/32	16										
. . .	0.025	0	S	1/16	10	111 800	82 100	96 600	111 400	92 600	7	11	3	4.74	0.87
		45	1/64	3/32	17										
		90	3/32	1/8	22	115 900	73 400	81 700	109 500	101 400	21	32	6		0.71
. . .	0.035	0	S	3/64	7	109 200	78 400	87 000	107 900	89 600	. . .	. . .	. . .	0.73.	0.80
		45	1/64	3/32	14										
		90	3/64	1/8	17										
. . .	0.045	0	S	3/32	13	102 100	79 100	86 700	102 700	101 600	17	25	4	5.29	0.85
		45	S	3/32	14										
		90	3/64	3/32	17										
. . .	0.120	0	S	3/32	13	94 100	72 200	80 500	90 900	90 300	11	20	3	5.49	0.86
		45	S	3/32	12										
		90	S	3/32	13										

(Continued on page 1090)

TABLE II.—TEST RESULTS.—*Concluded.*

Nominal Thickness, in.	Grain Size, mm.	Angle with Rolling Direction, deg.	Minimum Punch Radius, in.[a]		Springback, deg.	Tensile Strength, psi.	Proportional Limit, 0.002 per cent Offset, psi.	Yield Strength, psi.			Reduction of Area, per cent	Elongation, per cent		Erichsen Value, mm.	Ratio, Yield Strength to Tensile Strength
			Without Cracking	Without Orange Peel				at 0.01 per cent Offset	at 0.2 per cent Offset	at 0.5 per cent Strain		in 0.2 in.	in 2.0 in.		
HARD STRIP (CONDITION H)—*Continued*															
0.020	0.035	0	S	3/32	12	109 500	73 000	82 700	108 600	93 600	5	16	2	5.14	0.76
		45	1/64	1/8	16										
		90	3/32	5/32	22										
0.023	0.035	0	S	3/32	12	101 100	74 900	85 300	100 700	91 300	24	35	7	3.85	0.84
		45	1/64	1/8	15										
		90	1/32	5/32	18	104 900	59 800	73 800	104 100	94 800	31	44	9		0.70
0.027	0.035	0	S	3/32	10	106 100	70 900	81 500	105 100	91 000	20	31	5	5.51	0.77
		45	1/32	5/32	16										
		90	1/8	3/16	20	112 100	64 400	74 600	110 200	96 800	26	38	7		0.67
0.032	0.035	0	S	1/8	9	92 700	60 900	70 800	90 600	85 200	37	66	13	6.72	0.76
		45	S	5/32	14										
		90	S	3/16	14	97 100	58 600	66 700	94 100	82 800	51	64	12		0.69
0.040	0.025	0	S	3/32	8	108 600	77 500	86 500	107 500	92 900	29	48	7	7.11	0.80
		45	3/64	5/32	12										
		90	3/32	7/32	16	112 500	60 400	72 100	109 500	91 700	22	43	7		0.64
...	0.045	0	S	3/16	11	105 200	71 800	82 600	103 700	81 500	19	39	6	6.36	0.79
		45	3/64	1/4	15										
		90	5/32	1/4	17	110 100	63 300	75 600	108 800	94 300	38	56	9		0.69

[a] S = sharp.

creases in punch radius will lessen the tendency to produce orange peel.

Following bending, specimens were inspected with and without the aid of a microscope set to a magnification of 6.5×. Although orange peel frequently causes an objectionable appearance, Shanley (13) has reported, in the case of aluminum, that bending to the smallest possible radius does not adversely affect the fatigue properties if the material has not been cracked by the forming process.

Springback was also determined from these tests. The angle of the formed specimen was transferred to paper by an ink impression and measured. Springback is the difference between the measured angle and 90 deg. For angles over 90 deg., the springback is considered positive; for angles less than 90 deg., negative.

TEST DATA

Table II gives the test results for various tempers and includes bend test, springback, tensile and ductility data for various thicknesses, grain sizes and angles with rolling direction. Since each combination of nominal thickness and grain size shown includes from one to three lots of material, listed values are the averages of two to eight tests. For solution-treated and quarter hard tempers, results are listed for only one direction, since no directional effects were found. In the case of the other tempers, the omission of tensile results for specimens 90 deg. to the direction of rolling indicates that the strip was insufficiently wide for transverse specimens. Stress-strain and reduction-of-area determinations were not made on light gage stock.

The yield strength at 0.01 per cent offset is employed in determining the ratio of yield strength to tensile strength, since for hard strip the 0.2 per cent offset is frequently located at the same stress level as the ultimate. This latter condition would give a ratio of one, indicating no plastic deformation.

Table III gives a summary of tension test results for strip of 0.025 to 0.045 mm. grain size. For comparison, tensile strength and elongation values according to A.S.T.M. Specification B 194 – 49 T[3] are included.

TABLE III.—SUMMARY OF TENSION TEST RESULTS FOR STRIP OF 0.025–0.045 MM. GRAIN SIZE.

	Solution-Treated	Quarter Hard	Half Hard		Hard	
Angle with Rolling Direction, deg.	0	0	0	90	0	90
Tensile strength, psi.	63 400	78 500	85 800	89 400	106 800	111 000
Proportional limit (0.002 per cent offset), psi.	18 100	50 600	57 300	55 800	74 300	63 300
Yield strength, psi. (0.01 per cent offset)	22 000	58 100	65 500	65 400	84 400	74 100
(0.2 per cent offset)	30 500	71 200	83 500	89 000	104 200	106 000
(0.5 per cent strain)	32 000	70 900	79 900	85 900	91 000	93 600
Ratio, yield strength to tensile strength	0.35	0.73	0.76	0.71	0.80	0.68
Reduction of area, per cent	88	58	44	46	20	32
Elongation, per cent (in 0.2 in.)	81	59	48	48	27	40
(in 2.0 in.)	52	23	15	13	5	7
A.S.T.M. (B 194 – 49 T)						
Tensile strength, psi.	80 000 max.	73 000 min.	80 000 min.		95 000 min.	
Elongation in 2.0 in., per cent	35 min.	10 min.	5 min.		2 min.	

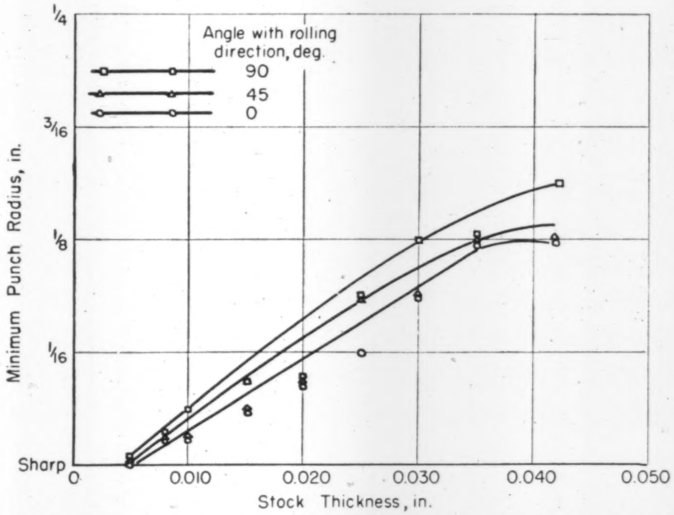

FIG. 1.—Minimum Forming Radii for Beryllium Copper Strip Rolled Half Hard.

The effect of stock thickness upon bending properties is presented graphically in Figs. 1 and 2. Since the curves in Figs. 1 and 2 are drawn through the extremes, points on the curves are not average values but actually approach the minimum forming radius from the safe side.

Gage length *versus* ductility as measured by elongation is shown in Fig. 3. Because of the extreme length (10 in.) of the specimens, it was possible to conduct only longitudinal tests.

Figures 4 and 5 illustrate the influence of punch radius and strip thickness upon springback. Springback data included in Table II result from measuring bend-test specimens used in determining the minimum punch radius without orange peel; consequently, the values shown apply only for the punch radii listed in the "without orange peel" column. The additional data included in Figs. 4 and 5 were obtained by measuring springback at various punch radii up to $\frac{1}{4}$ in.

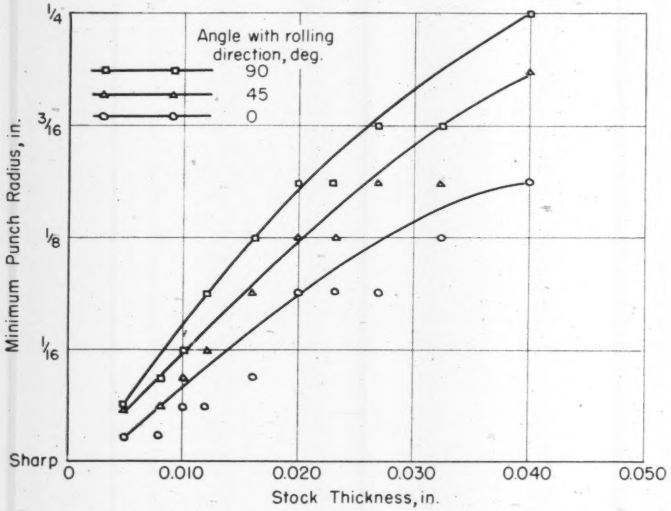

FIG. 2.—Minimum Forming Radii for Beryllium Copper Strip Rolled Hard.

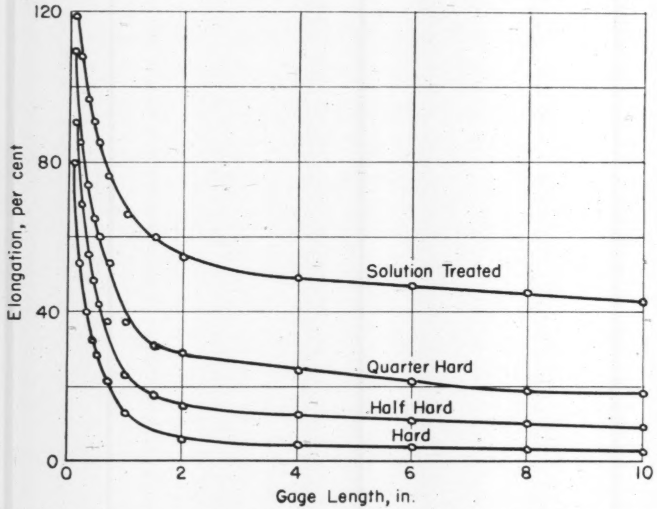

FIG. 3.—The Elongation of Beryllium Copper Strip 0.040 in. Thick of Various Gage Lengths.

Figures 6 and 7 plot depth of cup in the Erichsen test against stock thickness and grain size. The effect of grain size upon miscellaneous tensile properties is shown in Fig. 8, while Fig. 9 illustrates the influence of stock thickness upon elongation and reduction of area.

DISCUSSION OF DATA

Formability:

As indicated in Table II, solution treated and quarter hard strip up to

0.040 in. thick can be cold formed to a 90-deg. bend around a sharp radius without cracking or exhibiting orange peel. Although not listed in the tables, bend tests were conducted at 45 and 90 deg. as well as 0 deg. to the direction of rolling; however, no decrease in formability was noted. This was to be expected, since neither of these tempers revealed directionality during tension, elongation, or springback testing.

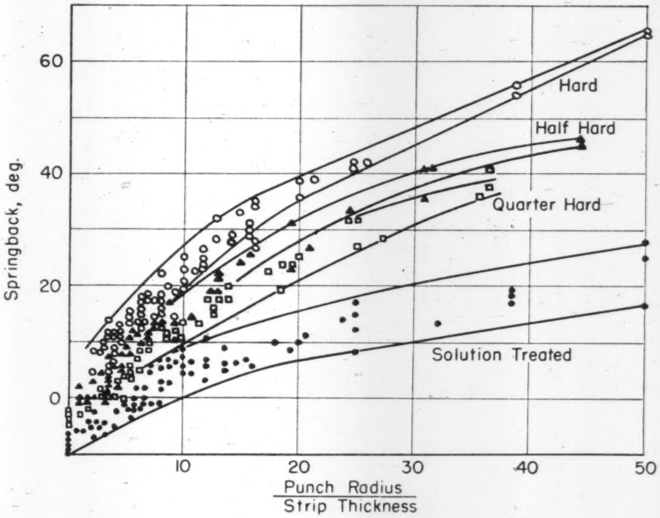

FIG. 4.—Springback of Beryllium Copper Strip.

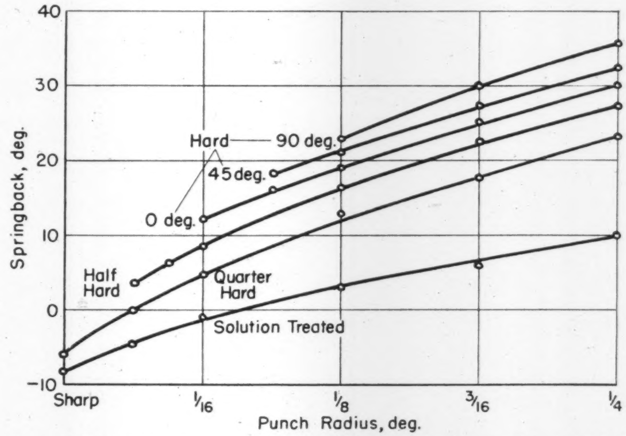

FIG. 5.—The Effect of Punch Radius upon Springback in Beryllium Copper 0.020 in. Thick Strip.

Figure 1 shows that there is a fairly linear relationship between minimum punch radius and stock thickness for half hard strip. The small degree of directionality revealed in bending this temper is confirmed by the slight increase in tensile strength listed for transverse specimens in Table III. Considerably greater directionality is apparent in the hard temper as evidenced by Fig. 2 and Table III. Although half hard material shows a definite decrease in formability with increased grain size, the results for the hard temper reveal no definite trend, except that directionality is apparently not a factor in larger grain sizes.

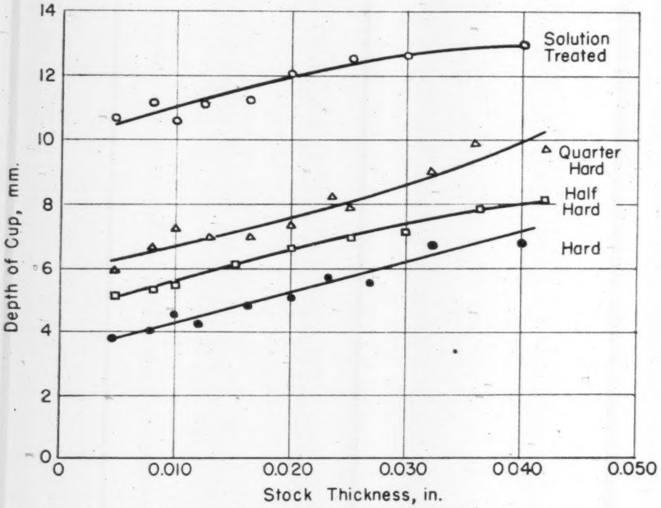

FIG. 6.—Erichsen Cup Test Values for Beryllium Copper Strip.

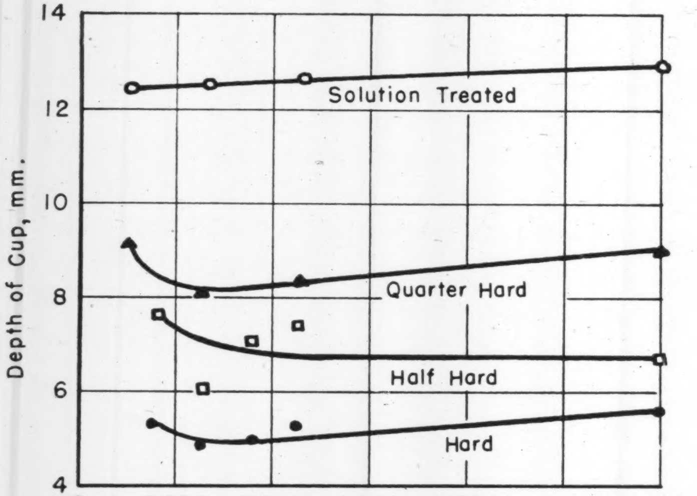

FIG. 7.—The Effect of Grain Size upon Erichsen Values for Beryllium Copper Strip.

Solution Treated—0.025 in. ¼ Hard—0.023 in.
½ Hard—0.020 in. Hard—0.016 in.

On the basis of tests conducted, no

simple relationship was found correlating bend-test results with tension and cup tests. According to Table III, the hard temper offers substantially higher reduction of area and elongation over 0.2- and 2-in. gage lengths for transverse than for longitudinal specimens; however, bend test results reveal better formability in the longitudinal direction.

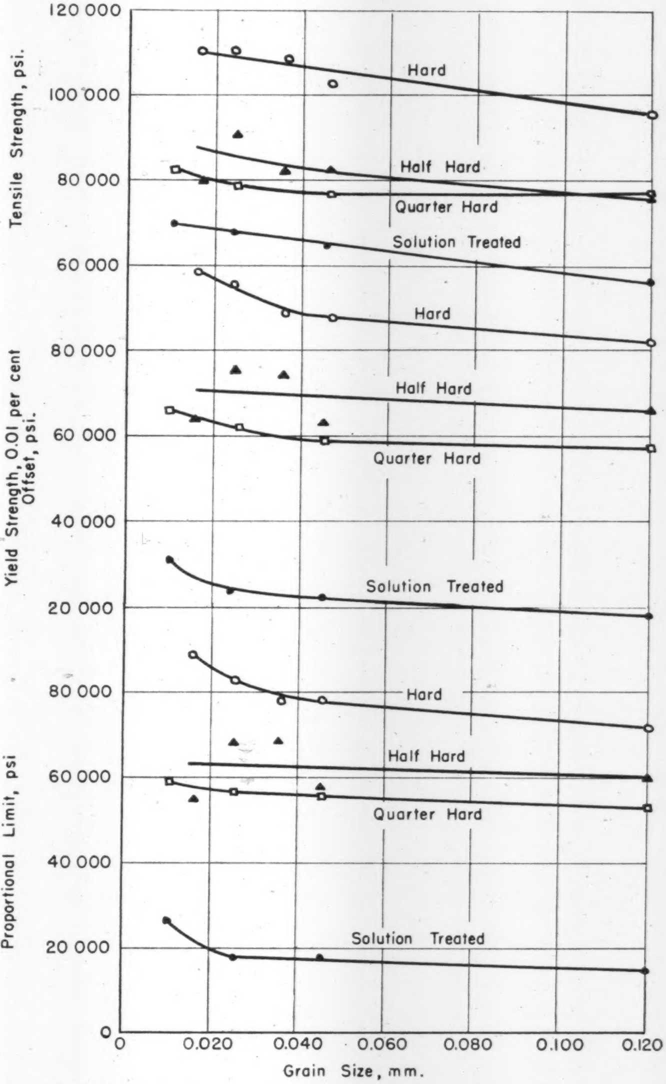

FIG. 8.—The Effect of Grain Size upon Tensile Properties of Beryllium Copper Strip.

During the tension testing of half hard and hard material, orange peel was first observed at the ultimate load and became more pronounced as the load dropped to the breaking point. For material of average grain size, orange peel was limited to the vicinity of the fracture and extended about 0.2 in. on either side of the fracture, roughly the length of the necked portion of the specimen. On the basis of this observation, it is possible to conclude that the elongation at the ultimate load approximates the elongation required to withstand bending without orange peel. This type is generally considered as uniform elongation as opposed to local elongation which occurs during necking. Although uniform elongation was not measured directly in this investigation, the elongation over 2 in. approximates the uniform elongation for the hard and half hard tempers according to Fig. 3; however, the test results, as previously indicated, show no correlation between formability and elongation over 2 in.

In a similar manner, elongations over short gage lengths should serve as a criterion for the minimum forming radius without cracking, since this takes into account localized elongation between the ultimate and breaking loads. This is the basis for correlation found between punch radius and elongation over short gage lengths observed for aluminum alloys (**5, 13, 14**). This approach to evaluating bending characteristics through elongation, however, apparently does not apply for the beryllium copper strip tested.

Figure 3 suggests that a greater variation in formability should be expected for bending without orange peel than without cracking, on account of the wider variation in elongation found for 2-in. gage length as compared to elongation over 0.2 in. for the several tempers tested. The bend-test data confirm this assumption, since hard material requires a greater punch radius than half hard, while solution-treated and quarter hard stock can be bent sharp without orange peel. On the other hand, the slight differences found in elongation over short gage lengths permit longitudinal specimens in all tempers to be formed to a sharp bend. The assumption that the 13-fold increase in elongation resulting

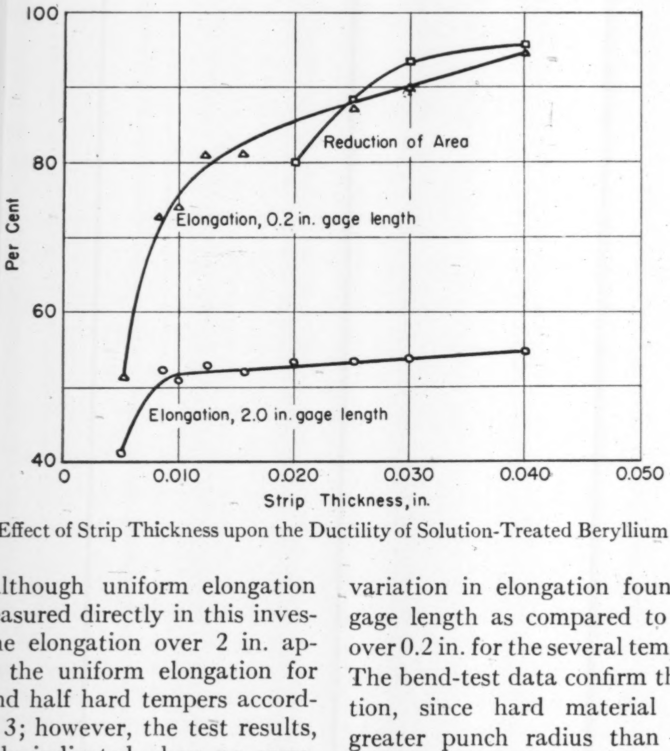

FIG. 9.—The Effect of Strip Thickness upon the Ductility of Solution-Treated Beryllium Copper Strip

from the decrease in gage length from 2 to 0.1 in. alone makes it possible to form hard material to a sharp bend is not valid. As shown in Table III, the transverse elongation of 0.2-in. gage length is greater than the longitudinal; however, the material cracks when bent sharp in the transverse direction.

Attempts to correlate bend-test results with Erichsen values or the ratio of yield strength to tensile strength were unsuccessful. A low ratio of yield strength to tensile strength may indicate a relatively large capacity for plastic deformation, and this relationship is found to hold in a general way, since the ratio increases with increasing temper. This agrees with similar tests on aluminum alloys by Templin (15), in which no reliable correlation was found between Erichsen values and tension or bending test values.

TABLE IV.—MINIMUM RADII FOR FORMING 90-DEG. BENDS IN BERYLLIUM COPPER STRIP.
For Heat-Treatable Material up to 0.040 in. Thick.

	Ratio, Punch Radius to Stock Thickness					
	Without Orange Peel			Without Cracking		
Angle with Rolling Direction, deg.	0	45	90	0	45	90
Solution-treated	0	0	0	0	0	0
Quarter hard	0	0	0	0	0	0
Half hard	3.0	3.5	4.0	0	0	0
Hard	4.5	6.0	7.5	0	1.5	3.0

Table IV summarizes the bending characteristics of the several tempers tested in various directions with respect to the rolling direction. These data are based on the minimum punch radius for forming a 90-deg. bend and apply for strip up to 0.040 in. thick. For convenience, formability is expressed as the ratio of punch radius to stock thickness.

Springback of bend test specimens was found to offer a reasonably accurate and sensitive index of forming and directional properties, as shown by Figs. 4 and 5. For a given thickness, springback increases with temper, punch radius and, in the case of harder tempers, with an increase in the angle with the direction of rolling. For a given punch radius, springback decreases with an increase in stock thickness.

Although Erichsen cup test values do not provide an accurate means for predicting formability, the results obtained reveal a fairly consistent increase in cup depth with an increase in stock thickness, as shown in Fig. 6. The effect of grain size upon the depth of cup (Fig. 7) is rather inconsistent, probably due to the relatively small number of grain sizes tested. It is apparent, however, that best results are attained with the smallest grain sizes, except in the case of solution-treated material.

Tensile Studies:

Tension tests were included in this project to determine anisotropic behavior and correlate tensile elongation with formability. Stress-strain curves were drawn, where possible, to obtain proportional limit and yield strength values. Results are summarized in Table III.

Directionality was checked by testing longitudinal and transverse specimens. Directional properties are apparently not a factor in solution-treated or quarter hard strip, since no significant differences were observed in tensile, elongation, or reduction-of-area values for specimens tested 0 or 90 deg. with respect to the rolling direction. This agrees with the results of bend and springback tests.

For half hard material, transverse specimens show a definite increase in tensile strength and yield strengths at 0.2 per cent offset and 0.5 per cent strain, a slight decrease in proportional limit, yield strength at 0.01 per cent offset, and yield strength-tensile strength ratio, and no appreciable change in elongation or reduction of area. These results tend to coincide with the slight directionality noted in bend and springback tests for this temper.

More pronounced directional characteristics are found in strip rolled hard. Tensile strength and yield strengths at 0.2 per cent offset and 0.5 per cent strain show a slight increase in the transverse direction, while elongation and reduction of area show a substantial (40 to 60 per cent) increase in this same direction. On the other hand, proportional limit, yield strength at 0.01 per cent offset, and yield strength – tensile strength ratio are considerably lower for transverse specimens. Here again, the directionality indicated by tension tests agrees closely with bend and springback results.

Apparently the directional properties of various copper alloys differ, although Gohn and Arnold (7) found that for 8 numbers hard silicon bronze and phosphor bronze transverse specimens give higher tensile strength and elongation with some loss of proportional limit. For many other copper alloys, the higher tensile strength in the transverse direction is accompanied by increased proportional limit and decreased elongation.

Figure 8, which plots tensile properties against grain size, indicates that an increase in grain size causes a drop in tensile strength, proportional limit, and yield strength. This decrease is more pronounced for the hard temper than for the others.

The limitations of tensile elongation as an indication of formability are well recognized. In a manner similar to other materials, the elongation of beryllium copper is greatly affected by the dimensions of the test specimen. Figure 9 shows that elongation and reduction of area fall off rapidly with a decrease in specimen thickness. At the same time a decrease in gage length causes an increase in elongation, particularly when gage lengths are less than 1 in.

SUMMARY AND CONCLUSIONS

Tests have been conducted to correlate the forming characteristics of beryllium copper strip with the results of conventional tension, elongation and Erichsen cup testing. Minimum safe forming radii for forming a 90-deg. bend were obtained by a direct method employing a punch-and-die set-up. The effects of temper, stock thickness, grain size, and grain direction upon formability, tensile strength, proportional limit, yield strength, elongation, reduction of area, and Erichsen values were determined.

The following conclusions can be drawn from this work:

1. Solution-treated and quarter hard beryllium copper strip can be bent sharp to a 90-deg. angle without cracking or displaying an orange peel effect.
2. Half hard and hard tempers can be bent to a 90-deg. angle without orange peel or cracking around moderate punch radii. The minimum forming radius varies directly with stock thickness.
3. No simple correlation was found relating formability with usual inspection or acceptance tests.
4. Directionality is absent from annealed and quarter hard material, slight in half hard strip and considerable in hard stock, as evidenced by bend and tension tests, elongation and springback.
5. Material having a relatively fine-grained structure offers the best combination of formability and tensile properties.

Acknowledgment:

The authors wish to thank The Beryllium Corp. for permission to publish these data. We are indebted to J. P. Butler for his helpful suggestions in the preparation of the manuscript.

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DISCUSSION

Mr. T. F. Olt.[1]—Will all the material you tested harden up to approximately the same level after the aging treatment?

TABLE V.—EFFECT OF GRAIN SIZE ON TENSILE PROPERTIES OF STRIP 0.010 IN. THICK.

	Solution-Treated (A)		Age-Hardened (AT)	
	0.010 mm. Grain Size	0.040 mm. Grain Size	0.010 mm. Grain Size	0.040 mm. Grain Size
Tensile Strength, psi...	69 300	59 300	173 900	160 300
Elongation in 2 in., per cent..................	29.5	40.0	5.5	1.5

TABLE VI.—EFFECT OF ROLLING DIRECTION ON TENSILE PROPERTIES OF STRIP 0.022 IN. THICK.

	Hard (H)			Age-Hardened (HT)		
Angle with Rolling Direction, deg..................	0	45	90	0	45	90
Tensile Strength, psi..............................	111 500	113 300	118 000	203 300	207 800	214 400
Proportional Limit, 0.002 per cent offset, psi........	77 600	63 200	54 100	148 700	193 900	196 500
Yield Strength, 0.2 per cent offset, psi..............	108 600	100 900	102 900	193 500	207 800	214 400
Elongation in 2 in., per cent.........................	4.0	4.3	6.0	2.5	1.7	1.3

Mr. J. T. Richards (*author's closure*).—The effect of precipitation hardening upon the variations in tensile properties resulting from directionality and different grain sizes is shown in the accompanying Tables V and VI. Values given are the averages for three tension tests.

Table V shows the influence of grain size on tensile strength and elongation for 0.010-in. thick strip in both the solution-treated (A) and solution-treated and precipitation-hardened (AT) conditions. This table indicates that a small grain size produces higher strength both before and after aging; however, a small grain size gives a lower elongation prior to aging and a higher elongation following aging.

In the case of directionality, a more complex situation results from precipitation hardening, as indicated in Table VI for 0.022-in. thick strip of 0.025 mm. grain size rolled hard (condition H). For specimens tested prior to aging, the tensile strength and elongation increase and the proportional limit and yield strength decrease for an increase in the angle with the rolling direction. Following precipitation hardening (condition HT) an increase in the angle with the rolling direction produces higher tensile strength, proportional limit, and yield strength with lower elongation.

Further investigations of the effect of precipitation hardening on directionality are in progress and will be reported at a later date.

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CHROMIUM-BASE ALLOYS*

By W. L. Havekotte,[1] C. T. Greenidge,[2] and H. C. Cross[2]

Prior to World War II, the metals that were used at high temperatures under stress were iron-, nickel-, and cobalt-base alloys. However, when the United States entered the war in December, 1941, better alloys were needed to withstand greater loads at higher temperatures—for example, for turbine blades rotating in an oxidizing atmosphere at temperatures up to 1600 F. (870 C.). The strongest high-temperature alloys available were cobalt-base materials. However, at that time cobalt was not plentiful, since most of it was imported and very little of it was stockpiled. Furthermore, the chances of improving the iron-, nickel-, or cobalt-base alloys to keep up with the development of gas turbines were not too good. Thus, one of the problems facing the metallurgist was how to make a better heat-resistant alloy without using cobalt.

In March, 1942, the War Metallurgy Committee of the National Defense Research Committee asked the Climax Molybdenum Co. to look into this problem. The investigators at Climax studied the chromium-rich regions of 13 binary and 9 ternary chromium alloy systems. Chromium-iron base alloys containing molybdenum, tungsten, or tantalum had the best combination of strength and ductility at 1600 F. and of shock resistance at room temperature. The Climax results[3] indicated that the most promising alloys for gas-turbine blades were the chromium-iron-molybdenum ternary alloys ranging in composition between 60 per cent chromium, 15 per cent iron, 25 per cent molybdenum and 60 per cent chromium, 25 per cent iron, and 15 per cent molybdenum. At this stage of development in September, 1945, the Office of Naval Research, Navy Department, sponsored a project at Battelle Memorial Inst. on "Alloys and Ceramics for High-Temperature Service." One phase of this project was a further study of chromium-base alloys. This program was aimed at obtaining chromium-base alloys with better combinations of strength and ductility than the Climax alloys had shown. In this program, it was proposed that the effects of improving the melting and casting technique, the effects of modifying the chemical composition slightly, and the effect of using various heat treatments be investigated. This paper is a condensed version of the final report[4] which summarized the results of research conducted at Battelle during the period from October, 1945, through March, 1950, on chromium-base alloys, particularly the 60Cr - 15Fe - 25Mo type alloy.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

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[2] Supervising Metallurgists, Battelle Memorial Inst., Columbus, Ohio.

[3] Robert M. Parke and Frederick P. Bens, "Chromium-Base Alloys," Symposium on Materials for Gas Turbines, p. 80, Am. Soc. Testing Mats. (1946). (Symposium issued as separate publication, *STP No. 68*.)

[4] W. L. Havekotte and C. T. Greenidge, "Chromium-Base Alloys," Final Report dated March 31, 1950, to Office of Naval Research, Department of the Navy (Contract N5ori-111).

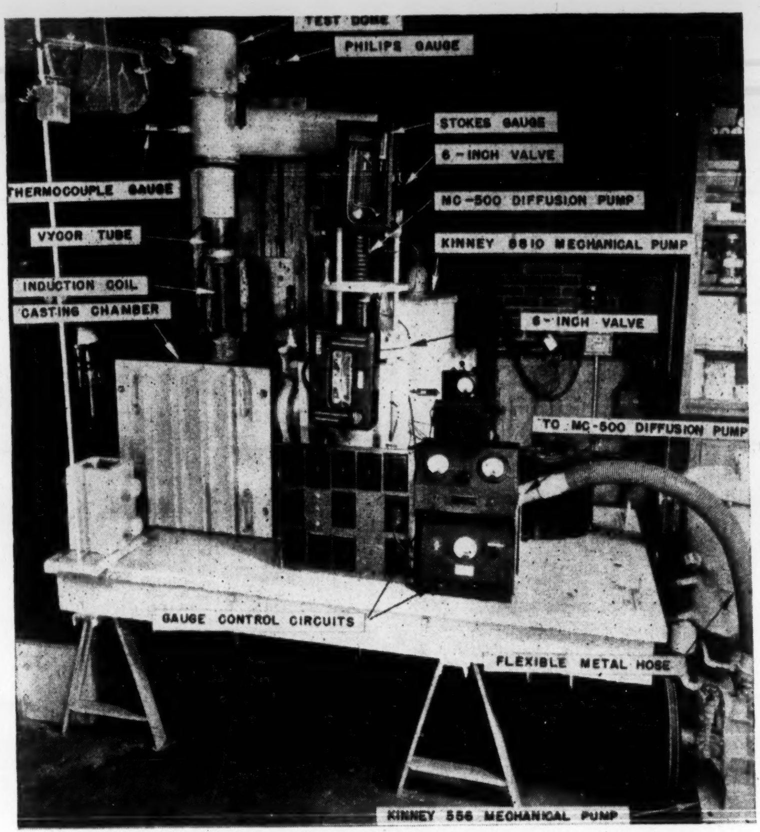

TEST DOME
PHILIPS GAUGE
STOKES GAUGE
6-INCH VALVE
THERMOCOUPLE GAUGE
MC-500 DIFFUSION PUMP
VYCOR TUBE
KINNEY 8810 MECHANICAL PUMP
INDUCTION COIL
CASTING CHAMBER
6-INCH VALVE
TO MC-500 DIFFUSION PUMP
GAUGE CONTROL CIRCUITS
FLEXIBLE METAL HOSE
KINNEY 556 MECHANICAL PUMP

FIG. 1.—Vacuum-Melting Apparatus.

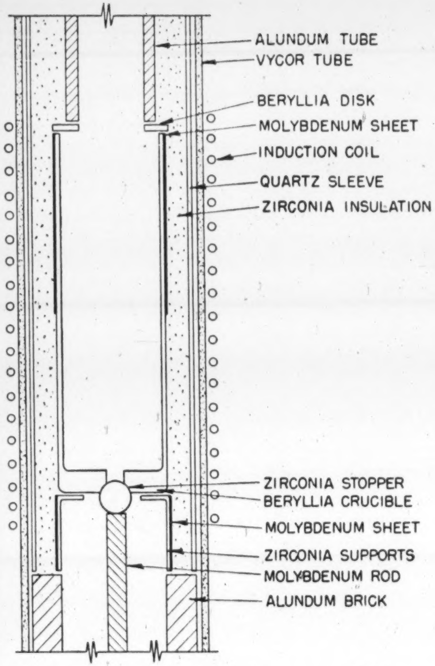
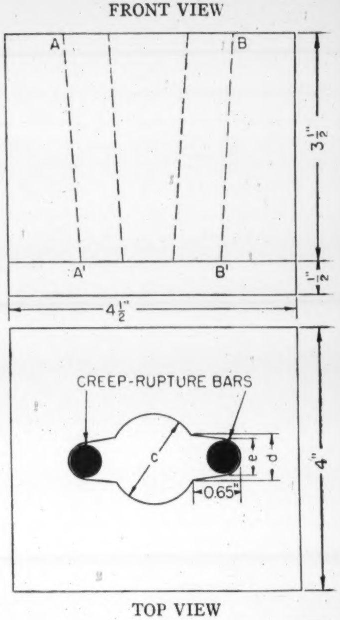

ALUNDUM TUBE
VYCOR TUBE
BERYLLIA DISK
MOLYBDENUM SHEET
INDUCTION COIL
QUARTZ SLEEVE
ZIRCONIA INSULATION
ZIRCONIA STOPPER
BERYLLIA CRUCIBLE
MOLYBDENUM SHEET
ZIRCONIA SUPPORTS
MOLYBDENUM ROD
ALUNDUM BRICK

FIG. 2.—Cross-Section of Vacuum-Melting Furnace.


FRONT VIEW
A
B
A'
B'
3 1/2"
1/2"
4 1/2"
CREEP-RUPTURE BARS
c
e
d
0.65"
4"
TOP VIEW

	AB	*A'B'*
c	1⅜ in.	1⅛ in.
d	0.60 in.	0.55 in.
e	0.55 in.	0.50 in.

FIG. 3.—Two-Wing Copper Mold.

MELTING AND CASTING APPARATUS

Several methods for preparing the chromium-base alloys were considered. From an examination of the various methods, it appeared that vacuum melting and casting was the most promising technique for producing chromium-base alloys of high quality and in the quantity desired for experimental work. Furthermore, a portion of the vacuum-melting and casting equipment at Climax was available for this study. Consequently, the Climax vacuum-melting and casting method was adopted and an apparatus similar to the Climax unit was designed and built. (A number of alloys were melted at atmospheric pressure under a protective slag covering. This method of preparing alloys is discussed more fully later in the paper.) Figure 1 shows the melting, casting, and vacuum equipment. Figure 2 is a cross-section of the melting furnace.

The melting equipment consists of a beryllia crucible of $3\frac{1}{4}$-lb. capacity held in the center of a 26-in.-long Vycor tube by insulating material. The induction coil is connected to an Ajax-Northrup 35-kw. spark-gap converter (approximately 30,000 cycles). The crucible is wrapped with a sheet of molybdenum. This sheet, heated by the induced current, heats the crucible uniformly and melts the charge without difficulty from bridging. A disadvantage of using a full-length molybdenum sheet heater is that the molten metal remains relatively quiet instead of being continuously stirred, as is the case when the charge is heated directly by induction. The use of a 4-in.-high molybdenum sheet heater around the top half of the beryllia crucible prevented bridging of the charge and allowed some stirring of the melt.

Beryllia crucibles have shown an average life of 4 to 6 melts and a maximum life of 15 melts. The satisfactory performance of beryllia in this application is attributed to its high thermal conductivity, good thermal-shock resistance, and freedom from an allotropic transformation.

Measurements of the melt temperature were made using an optical pyrometer. This was sighted through a hole at the top of the furnace that was normally closed by an easily removable steel ball. The steel ball protected the glass viewing plate from metal spatter and fumes and permitted an accurate reading of the melt temperature. The 60Cr - 15Fe - 25Mo type alloy melts at about 3050 F. and is tapped at 3250 to 3400 F. Temperatures in the range of 3750 to 3800 F. have been obtained with this unit.

A late alloy addition can be introduced into the melt through a rubber tube, suspended above and to one side of the top of the furnace. Late additions of aluminum, manganese, titanium, zirconium, silver, tungsten, boron, and selenium were made in this manner.

After each heat is poured, the furnace and casting chambers must be opened so that the mold may be replaced, the crucible recharged, and the stopper reset in the base of the crucible. The system must then be re-evacuated before melting can start.

With the above-described set-up, a heat is tapped from the crucible by withdrawing a zirconia-ball stopper plug, which seals the hole in the bottom of the crucible, and allowing the molten metal to run out under the force of gravity. The zirconia plug is held in place by a molybdenum stopper rod (Fig. 2). This rod, actuated by a solenoid to which it is attached, is pulled down and away from the hole, and the alloy flows into the mold. The pressure at the time of tapping was usually below 8 μ of mercury.

The mold designs and materials were changed as the work progressed so as to improve the soundness and the grain structure of the castings. Heats were tapped into copper molds with round-cornered-square, and two- and three-winged cavities, all tapered; into cast iron molds with round and two-winged tapered cavities, and with copper stools; and into stainless steel and zircon refractory two-wing tapered molds. Progressive changes in mold design and in materials almost entirely eliminated microporosity in the winged sections of the castings. The design of a typical mold is shown in Fig. 3. One creep-rupture bar was obtained from each wing section of the casting made in this mold. The center portion of the casting acted as a feeding section for the wings during solidification. A refractory combination funnel and hot top was placed on the top of the mold to assist in pouring and feeding the casting.

The vacuum equipment used and shown in Fig. 1 is described below.

Connected in series to the top of the melting unit were a Mc-500 Distillation Products Co. oil-diffusion pump, and a Kinney No. 8810 mechanical pump with a capacity of 110 cu. ft. per min. Connected in series to one side of the casting chamber were a Mc-500 Distillation Products Co. oil-diffusion pump, and a Kinney No. 556 mechanical pump with a capacity of 13 cu. ft. per min. Two Stokes-McLeod gages, one Phillips gage, and five thermocouple gages were used to measure the pressure at different parts of the system.

A helium-probe, mass-spectrometer leak detector was used for locating the leaks in the vacuum equipment. After the system was made practically leak free, the rate of inleakage and the pumping speed of the equipment were determined. Tests showed that the rate of inleakage for this system was approximately 1.6 μ liters per sec. at 0.1 μ pressure. The pumping speed of the equipment (Fig. 4) was determined using the method described by Dayton.[5] The test dome, used only for determining pumping speeds for the vacuum-melting equipment, can be seen in Fig. 1. Preparatory to melting, the test dome was replaced by the head which contained a viewing window and equipment for making late alloy additions.

PREPARATION OF TEST MATERIALS

Melting and Casting in Vacuum:

The melting stock generally consisted either of low-carbon (under 0.10 per cent), low-nitrogen (under 0.10 per cent), ferrochromium (70 per cent chromium), low-carbon (0.04 per cent), low-nitrogen (0.011 per cent), fused-chromium metal (99.2 per cent chromium), molybdenum powder, and carbon; or of electrolytic chromium, electrolytic iron or Puron,[6] molybdenum powder and carbon. When the carbon content of the casting was to be less than 0.05 per cent, the latter type of charge was used. It was found advisable to add the molybdenum as powder to the bottom two-thirds of the charge. Otherwise, during evacuation of the apparatus, there was a tendency for some of the powder to be pulled out of the charge.

Several melting procedures were used. The procedure stated below was found to be the most satisfactory:

1. Heat the charge (power input 10 to 14 kw.) to about 2600 F. in about 45 min.
2. Continue to heat (power input 18 kw.) until the charge is molten and the temperature of the melt is 3400 to 3450 F.
3. Maintain the temperature of the alloy at 3400 to 3450 F. for 5 min.
4. Decrease the temperature of the alloy to about 3275 to 3325 F.
5. After the alloy has been molten for 30 min., tap the heat.

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[5] B. B. Dayton, "Measurement and Comparison of Pumping Speeds," *Industrial and Engineering Chemistry*, Vol. 40, May, 1948, p. 795.

[6] High-purity iron supplied by Westinghouse Electric Corp., E. Pittsburgh, Pa.

In general, when the alloy was cast into zircon refractory molds, the casting was cooled slowly in the mold to room temperature. When metal molds were used, the castings were removed from the molds 20 min. after tapping, heated to 1200 F. and held at temperature for 3 hr., and then furnace-cooled to room temperature. These treatments reduced the tendency for the castings to crack. Cooling to room temperature in metal molds resulted in cracking.

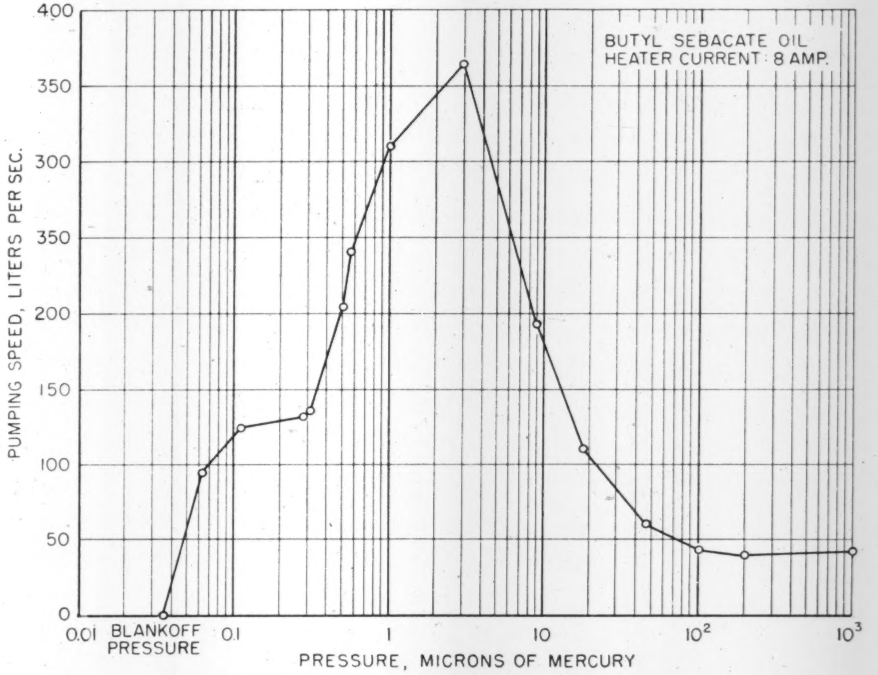

Fig. 4.—Pumping Speed Curve for Vacuum-Melting Furnace.

Melting and Casting at Atmospheric Pressure:

A number of chromium-base alloy heats were induction melted at atmospheric pressure in magnesia crucibles under slags containing 40 per cent lime, 50 per cent alumina, 10 per cent magnesia. These alloys were prepared to test the suitability of various investment mixes for precision casting chromium-base alloys as turbine blades. Air melting is more rapid than vacuum melting and, consequently, expedited the evaluation of the investment materials.

Tests were conducted on the casting of the 60Cr - 15Fe - 25Mo type alloy in refractory molds prepared using most of the commercially available investments (refractories and binders). The melting stock for melts under slag generally consisted of low-carbon, low-nitrogen fused chromium metal, electrolytic iron or Puron, molybdenum bar stock, and carbon. Most of these alloys were deoxidized with 0.1 per cent calcium added as calcium silicide. Carbon was not an efficient deoxidizer for air-melted alloys. All of the carbon added to the charge was recovered in the casting. Castings with smooth surfaces were not obtained. Zircon-base (zirconium silicate) or beryllia-base investments might be satisfactory if used with a binder which did not react with the molten metal. However, such a binder was not found.

Tests were also made at the Works Laboratory of the General Electric Co., and at the Mercast Corp. on casting the 60Cr - 15Fe - 25Mo type alloy in the form of TG-190 turbine blades in zircon-base investment molds. Mercast[7] used the frozen-mercury-pattern process in preparing the molds. The tests conducted there produced the first chromium-base alloy blades that did not crack on cooling to room temperature. The A.S.T.M. grain size[8] of the alloy near the leading edges of the blades was No. 6 to 7. The surface finish on these blades was fair but not good enough to satisfy present-day requirements. However, the blades were smoother than any prepared previously. It is suspected that, because of the high pouring temperature of the chromium-base alloy, the molten metal reacted with the investment binder and this reaction produced rough surfaces on the castings.

On several occasions the air-melted alloy was also poured into copper or refractory molds to prepare creep-rupture bars. These alloys were not so strong as those prepared by vacuum melting and casting, but their performance was encouraging.

Two melting procedures were tried to determine the amount of nitrogen picked up during melting. When the major portion of the charge was added to the melting crucible and the slag was placed on top of the cold charge, the cast metal had a high nitrogen content, averaging about 0.34 per cent. The nitrogen content of the alloy was about the same when no slag was used. When low-carbon, high-nitrogen ferrochromium was charged without using a slag covering, the nitrogen content of the casting was 0.83 per cent. When chromium and molybdenum were added gradually to a pool of molten iron with the slag covering the metal at all times, the resultant ingot had a much lower nitrogen content, averaging 0.056 per cent. This was only slightly higher than the nitrogen content of a similar alloy prepared in the vacuum apparatus.

Although most of the chromium-base alloy heats were prepared by vacuum melting, enough heats were melted in air under a slag to indicate that air melting might be developed into a commercial method of producing these alloys. This would be important in an emergency, because there probably would not be enough vacuum equipment to produce chromium-base alloys on a large scale.

Chemical Composition:

Most of the alloys discussed here are of the 60Cr - 15Fe - 25Mo type, with and without 2 per cent titanium. The chemical compositions for these alloys are listed in Table I. Also indicated are the compositions of the 60Cr - 15Fe - 25Mo type alloy made with additions of silver, tantalum, beryllium, nitrogen, tungsten, and boron.

To study the effects of composition variations, several alloys were prepared with chromium contents ranging from 54 to 68 per cent, various combinations of iron and molybdenum, and a constant titanium content of 2 per cent. These alloys showed no improvement in 1600 F. rupture properties over those of the 60Cr - 15Fe - 25Mo type alloy. Substitution of manganese, cobalt, or nickel for iron, and of tungsten for molybdenum was also tried. The addition of manganese was beneficial in promoting consistency in creep-rupture properties at 1600 F. The cobalt- and nickel-bearing

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[7] Herbert Chase and Leslie Schakenback, "New Precision Casting Process Provides Better Finish and Closer Tolerances," *Materials & Methods*, March, 1949, p. 52.

[8] Standard Classification of Austenite Grain Size in Steels, A.S.T.M. Designation (E 19 - 46), 1949 Book of A.S.T.M. Standards, Part 1, p. 1271.

TABLE I.—CHEMICAL COMPOSITIONS AND MELTING DATA.

Heat	Chemical Composition, per cent[a]								Melting Data			ASTM Grain Size Number
	Chromium	Iron	Molybdenum	Carbon	Silicon	Nitrogen	Oxygen	Titanium	Casting pressure, μ	Casting Temperature, deg. Fahr.	Mold Temperature, deg. Fahr.	
A-3851	58.9	15.5	23.0	0.12	0.51	0.034	0.022	1.63	58	3180	80	6 to 7
A-4082	57.8	15.8	25.5	0.06	0.57	0.022	0.010	...	250	3320		0 to 1
A-4083	57.9	15.9	25.5	0.07	0.58	0.030	0.007	...	32	3380		3
A-4084	58.5	15.7	25.1	0.07	0.55	0.018	0.007	...	20	3300		3
A-4085	59.5	15.2	24.4	0.08	0.56	0.024	0.010	...	45	3360		3 to 4
A-4095	58.7	15.2	23.8	0.11	0.51	0.056	0.024	1.81	15	3210	80	5 to 6
A-4253	57.8	15.5	24.2	0.11	0.62	0.043	0.012	1.94	277	3280		3
A-4254	57.9	15.2	24.2	0.13	0.61	0.041	0.012	1.80	128	3270		3 to 4
A-4255	58.9	15.3	24.8	0.10	0.59	0.037	0.017	1.70	158	3260		4
A-4256	58.3	14.9	23.6	0.11	0.62	0.056	0.017	1.93	125	3260		4
A-4257	58.1	15.0	24.4	0.12	0.62	0.042	0.014	1.78	143	3260	80	3 to 4
A-4276[b]	58.9	(14)	(24)	0.03	(0.51)	0.048	...	(2)	...	3180		3
A-4277[b]	59.4	(14)	(24)	0.06	(0.51)	0.059	0.033	(2)	...	3260		3 to 4
A-4566	56.0	16.0	25.9	0.12	(0.54)	0.049	0.008	2.03	37	3300		7
A-4567	58.3	15.2	24.5	0.09	(0.54)	0.046	0.006	2.01	47	3250		4
A-4568	57.7	15.6	24.8	0.11	(0.54)	0.042	0.010	2.05	200	3350	80	5
A-4569[b]	58.8	(14)	(24)	0.05	(0.51)	0.062	0.024	(2)	...	3240		4 to 5
A-4685	67.2	11.3	19.0	0.08	0.53	0.032	0.016	2.04	333	3250		2 to 3
A-5153	60.9	13.8	(23)	0.12	(0.47)	0.022	0.022	1.84	162	3180		6
A-5305[b]	59.3	14.2	(24)	0.03	(0.53)	0.062	0.020	(2)	...	3140		4
A-5307[b]	58.9	14.6	(24)	0.04	(0.53)	0.057	0.022	(2)	...	3300	80	4
A-5314	58.7	14.4	24.2	0.02	(0.48)	0.033	0.016	1.63	327	3350	80	4
A-5811	56.3	15.2	26.4	0.16	0.10	0.009	0.021	1.6	3	3410	1200	5
A-5818	60.8	13.9	23.4	0.05	0.17	0.007	0.042	1.5	11	3410	1200	3
A-5819	55.1	15.1	27.1	0.06	0.17	0.010	0.030	1.7	6	3420	1200	3
A-5820	55.2	15.1	27.7	0.03	0.13	0.008	0.034	1.6	6	3400	1200	4 to 5
A-5821	55.5	15.6	27.0	0.12	0.07	0.009	0.014	1.6	8	3420	900	3 to 4
A-5822	55.3	15.5	26.9	0.13	0.10	0.023	0.012	1.5	9	3410	600	4 to 5
A-5823	54.8	15.2	27.3	0.15	(0.10)	0.010	0.023	1.8	8	3420	300	5 to 6
A-5840	54.2	16.5	29.4	0.01	(0.01)	0.011	0.152	...	2	3100	600	5
A-5841	60.5	14.5	23.2	0.05	(0.01)	0.015	0.027	1.67	26	3420	1200	0
A-5843	58.7	14.8	24.8	0.08	(0.01)	0.017	0.015	1.91	6	3410	600	1
A-5844	55.8	16.1	25.9	0.05	(0.01)	0.016	0.020	1.87	3	3420	80	1
A-5845	62.0	13.6	23.4	0.02	(0.01)	0.007	0.202	...	110	3400	1200	−1
A-5846	55.4	15.6	28.4	0.04	(0.01)	0.011	0.193	...	80	3420	600	−2
A-5847	60.9	14.5	23.9	0.02	(0.01)	0.013	0.191	...	135	3420	80	−2
A-5848	57.7	14.7	24.5	0.04	(0.44)	0.030	0.013	2.07	2	3400	1200	2
A-5849	57.3	14.6	24.9	0.04	(0.44)	0.031	0.014	2.13	2	3420	600	2 to 3
A-6078	59.4	14.3	23.4	0.07	0.48	0.028	0.010	2.16	3	3280	80	4
A-6079	61.4	14.8	23.6	0.03	0.04	0.006	0.206	...	1	3250	80	2 to 3
A-6085	59.9	14.1	23.8	0.10	0.48	0.039	0.014	1.94	21	3310	80	4 to 5
A-6086	59.3	14.4	24.3	0.07	0.46	0.024	0.014	1.83	5	3300		3
A-6087	56.1	15.4	28.4	0.03	(0.01)	0.003	0.21	...	1	3110		3 to 5
A-6088	58.9	15.4	25.5	0.07	(0.01)	0.005	0.14	...	1	3310		2 to 4
A-6089	58.8	15.3	26.0	0.01	0.07	0.006	0.16	0.04B	1	3250		4 to 5
A-6090	54.3	16.3	29.1	0.01	0.02	0.004	0.14	0.1B	1	3260	80	3 to 4
A-6091	57.5	15.0	27.1	0.01	0.02	0.005	0.061	0.5B	1	3280		4 to 5
A-6092	59.5	12.3	23.9	0.01	0.12	0.016	0.018	2.8B	1	3260		...
A-6093[b]	58.3	15.5	24.2	0.02	0.59	0.36	0.24	...	...	3240		0
A-6094[b]	59.6	14.7	23.2	0.04	0.57	0.86	0.24	...	...	3200		0
A-6095	59.8	14.3	23.2	0.04	0.53	1.48	0.18	...	...	3140	80	...
A-6096	55.0	15.4	28.9	0.04	0.04	0.006	0.18	Nil Ag	1	3320		3 to 4
A-6099[b]	59.2	14.3	24.4	0.02	0.48	0.35	0.076	0.04 Ta	...	3250		3 to 5
A-6400[b]	59.0	14.5	24.7	0.02	0.55	0.24	0.033	0.08 Be	...	3280		0 to 2
A-6402	55.8	15.6	28.3	0.02	0.04	0.007	0.24	0.06 W	1	3270		3 to 5
A-6403	57.2	15.5	27.1	0.02	0.04	0.011	0.27	0.54 W	1	3290	80	3 to 4
Stellite No. 21	(26.2)	...	(5.7)	(0.24)	(0.7)	...	(2.7)Ni	Bal. Co	...	...	...	...
Stellite No. 31	(25)	...	(8)W	(0.50)	(0.7)	...	(10)Ni	Bal. Co	...	...	...	...
Cast Iron	...	Bal.	...	3.75	1.98	...	...	0.7Mn	...	...	...	...

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[a] The values in parentheses refer to intended and not actual compositions.

[b] Alloy was melted at atmospheric pressure under slag.

NOTE.—Unless noted to the contrary, the alloys were melted in vacuum.

alloys were so hard and brittle that tests bars could not be ground. Tungsten refined the grain size slightly; however, the tungsten-bearing alloys broke on loading.

Up to 4 per cent of vanadium or columbium was added to replace titanium in the 58Cr - 15Fe - 25Mo - 2Ti type alloy. Vanadium had no effect on the rupture properties. The addition of columbium resulted in a slight grain coarsening. At the time the columbium-bearing alloys were made, chromium-base alloys with an A.S.T.M. grain size of No. 2 or larger usually yielded poor creep-rupture properties. Consequently, the columbium-bearing alloys were not tested.

The addition of manganese plus titanium to the 60Cr - 15Fe - 25Mo type alloy produced large columnar grains in the casting. Creep-rupture bars of the alloys broke on loading.

Test Specimens:

In preparing test specimens, coupons were removed from the castings by means of a cut-off wheel and ground to size. Because of the poor machinability of the 60Cr - 15Fe - 25Mo type alloy, it was expedient to prepare the test specimens by grinding. Since this alloy cracks readily, the specimens were cut and ground with care.

Creep-rupture bars were taken from the wing sections of the castings. These were 3 in. in over-all length, having $\frac{1}{2}$-in.-diameter threaded ends, $\frac{1}{4}$-in.-diameter reduced sections, and 1-in.-long gage lengths. Before testing, the bars were examined for flaws by the Zyglo test.

Alloys for bend and impact testing were cast in refractory molds. Each mold contained seven bars, each of which tapered from $\frac{3}{8}$ by $\frac{1}{2}$ in. to $\frac{1}{2}$ by $\frac{1}{2}$ in. in $3\frac{1}{2}$ in. Bend-test specimens, 3 in. long by 0.31 in. wide by 0.2 in. thick, and impact specimens 2.15 in. long by 0.34 in. square were ground from these bars.

In the study of the aging characteristics of this alloy, specimens approximately $\frac{1}{2}$ in. square by $\frac{1}{4}$ in. thick were taken from the sprue sections of the castings. The specimens were heat treated in air and Vickers hardness measurements (10-kg. load) were made.

For the oxidation tests, specimens 1.00 in. long by 0.375 in. diameter were ground from the wing sections of the 60Cr - 15Fe - 25Mo type alloy (heats A-4082 through A-4085).

The specimens used in the thermal-shock tests were ground from a four-bar casting made in a refractory mold. These were wedge shaped, tapering from $\frac{1}{4}$ in. to $\frac{1}{32}$ in., and 2 in. long by $1\frac{1}{4}$ in. high.

MICROSTRUCTURE

Metallographic examination of the 60Cr - 15Fe - 25Mo type alloy, containing 0.05 to 0.10 per cent carbon and less than about 0.05 per cent of nitrogen, indicated that the following three phases were present[9]:

1. Chromium-rich solid solution. This is the matrix phase and has a body-centered-cubic lattice.

2. As-cast and precipitated carbides. These are identical and are a complex face-centered-cubic $M_{23}C_6$-type carbide, indicated by X-ray data to be $(Cr, Mo)_{23}C_6$. The block-sample focusing-camera method and the powder-diffraction method on aggregate specimens and on electrolytically separated residues were used in identifying the carbide. The carbide formed during casting is located chiefly at the grain boundaries, while a small amount is found within the grains. The carbide appears to be the lowest melting-point constituent found

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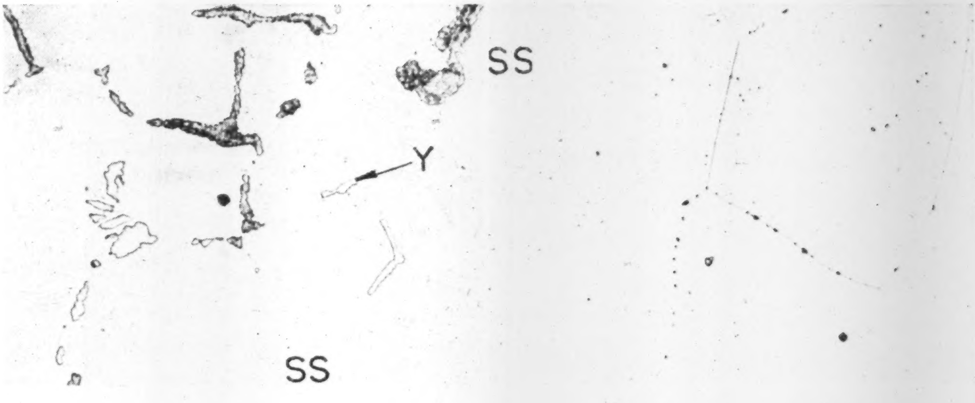
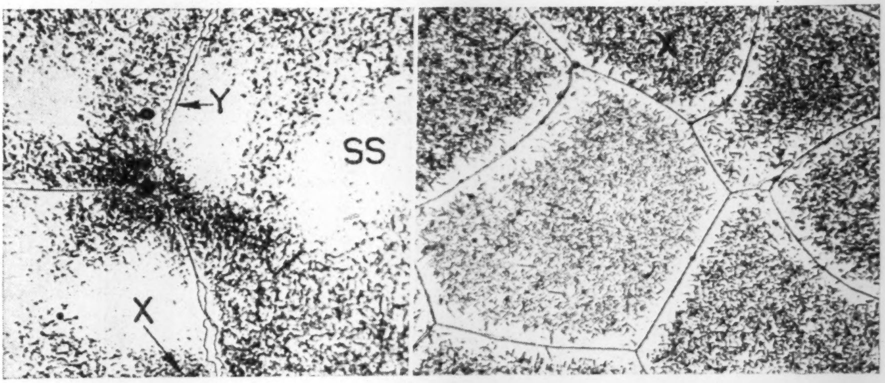
[9] J. P. Hammond, "An Investigation of the Microconstituents in Chromium-Base, Chromium-Iron-Molybdenum Alloys and Their Behavior with Heat Treatment." Doctorate dissertation on file at Battelle Memorial Institute and the Engineering School of Yale University (1949).



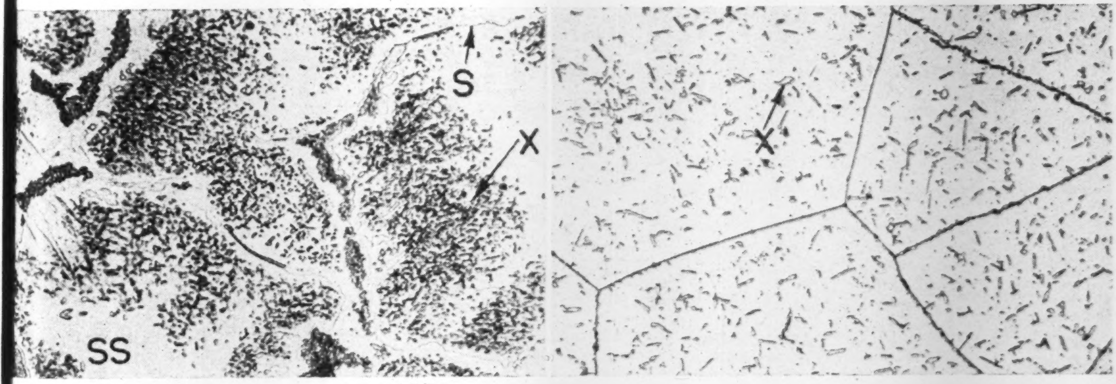
(*a*) Cast and Stress-Relieved.

(*d*) Cast, Stress-Relieved, and Solution-Treated at 2500 F. for 50 hr.



(*b*) Cast, Stress-Relieved, and Aged at 1600 F. for 96 hr.

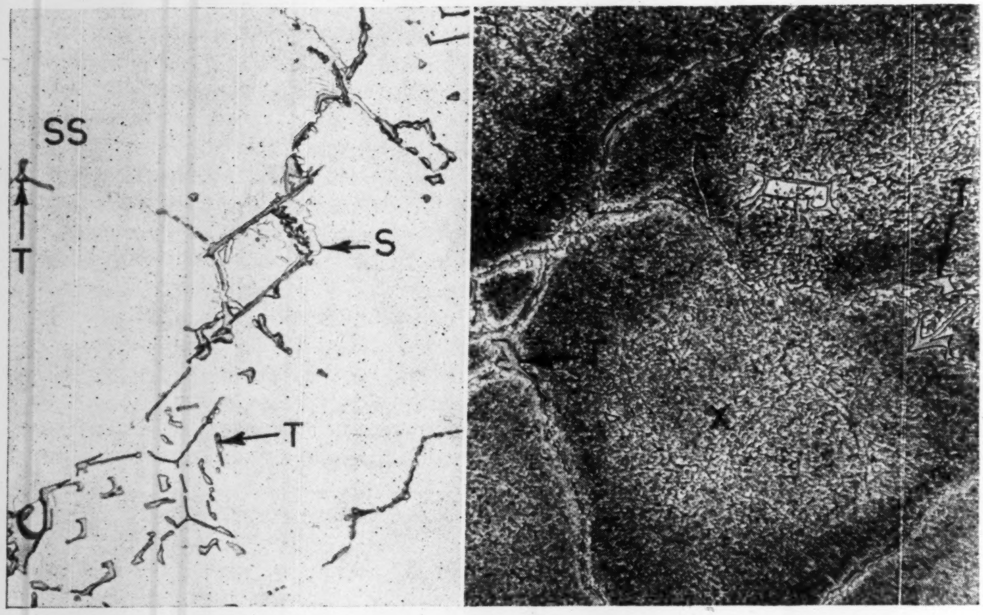
(*e*) Cast, Stress-Relieved, Solution-Treated, and Aged at 1600 F. for 96 hr.



(*c*) Cast, Stress-Relieved, and Aged at 1800 F. for 96 hr.

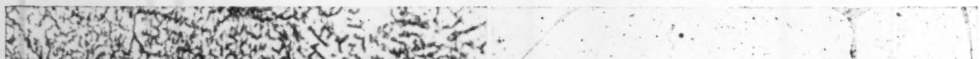
(*f*) Cast, Stress-Relieved, Solution-Treated, and Aged at 1800 F. for 96 hr.

FIG. 5.—Microstructures of 60Cr - 15Fe - 25Mo - 0.08C Alloy (Heat A-4085) (×500).



(*a*) 58Cr - 15Fe - 25Mo - 2Ti - 0.13C Alloy. Cast and Stress Relieved (× 500).

(*b*) 58Cr - 15Fe - 25Mo - 2Ti - 0.13C Alloy. Creep-Rupture Bar Tested at 1600 F. and 38,000 psi. for 760 hr. (× 500).



(*c*) 60Cr - 15Fe - 25Mo - 0.05C Alloy. Creep-Rupture Bar Tested at 1600 F. and 30,000 psi. for 1441 hr. (× 1000).

(*d*) 60Cr - 15Fe - 25Mo - 0.05C Alloy. Heated at 2700 F. for 3 hr. (× 100).

FIG. 6.—Microstructures of Chromium-Base Alloys.

in this alloy. There are indications that it melts at temperatures above 2500 F. The precipitated carbide is found in specimens aged at 1600 or 1800 F. It appears as a very fine precipitate adjacent to the grain boundaries and in the interstices of the dendrites. On long-time aging at 1600 or 1800 F., the precipitate coalesces into large particles.

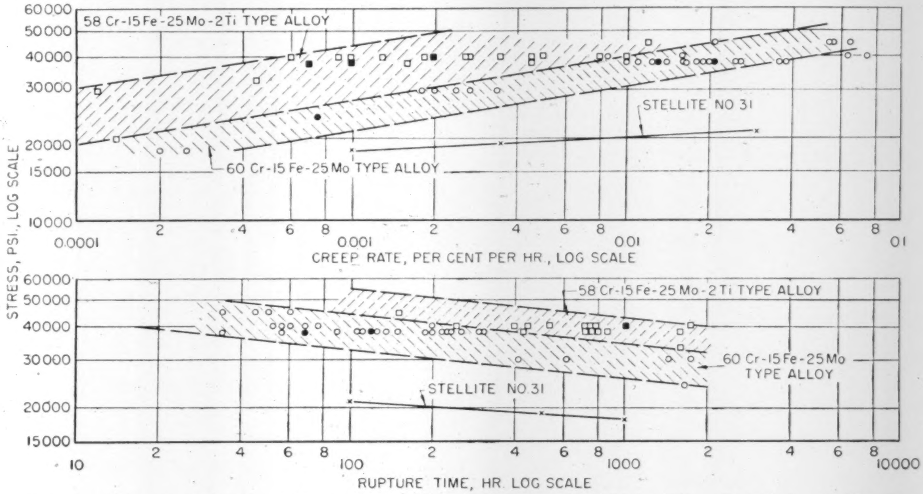


FIG. 7.—Rupture-Time and Creep-Rate Data for Chromium-Base Alloys Tested at 1600 F.

3. Sigma phase. This is identical with the sigma phase found in chromium-iron binary alloys; X-ray studies show it to be isomorphic with the FeCr intermetallic compound of the chromium-iron binary system. Sigma forms as massive particles next to the as-cast carbide. Sigma appears in the microstructure of the 60Cr - 15Fe - 25Mo type alloy heated at 1600 or 1800 F. and in the as-cast structure of this type of alloy containing 2 per cent titanium.

In alloys containing 2 per cent titanium, face-centered-cubic titanium carbide and a solid solution, composed of this carbide and titanium nitride, were observed.

According to Hammond[9] a constituent of undetermined composition, "Z" phase, is found in an alloy containing 0.01 per cent carbon. This manifests itself in a finely dispersed form.

Figures 5 and 6 illustrate typical structures of the chromium-base alloys. All specimens were electrolytically etched in a solution of 100 ml. alcohol, 20 ml. nitric acid, and 20 ml. ethylene glycol. The phases present in amounts large enough to be seen in the micrographs are identified as follows:

KEY	PHASE
SS	Chromium-rich solid solution
Y	As-cast $M_{23}C_6$-type carbide
X	Precipated $M_{23}C_6$-type carbide
S	Sigma
T	Solid solution of titanium carbide and titanium nitride

The dark areas at the grain boundaries in Fig. 5(*a*) have a eutectic structure, which at 1000 magnification cannot be positively identified. The two constituents are thought to be solid solution and carbide. The eutectic structure is not always observed in this type of alloy; the reason for its presence or absence is not known. The black particles shown in Fig. 5(*d*) are oxide inclusions; the oxygen was picked up superficially during the solution treatment. Figure 6(*d*) reveals melting of the $M_{23}C_6$-type carbide at the grain boundaries.

TABLE II.—CREEP-RUPTURE RESULTS FOR CHROMIUM-BASE ALLOYS AT 1600 AND 1800 F.

Specimen Number	A.S.T.M. Grain Size Number	Creep-Rupture Data							
		Stress, psi.	Rupture Time, hr.	Elongation, per cent	Reduction of Area, per cent	Minimum Creep Rate, per cent per hr.	Hours for Total Deformation of		
							1 per cent	2 per cent	5 per cent
Tested at 1600 F.									
A-3851-2*	6 to 7	20 000	1416+	0.8	...	0.00014	...	...	...
A-4254-2*	3 to 4	30 000	1654+	...	...	0.00012	1400	...	...
A-5153-2*	6	33 000	1613	3.2	Nil	0.00045	90	1350	...
A-4253-1*	3	38 000	864	1.5	0.8	0.0007	300	...	...
A-4257-1*	3 to 4	38 000	721	1.6	1.6	0.0010	230	...	...
A-4254-1*	3 to 4	38 000	761	2.4	2.4	0.0007	160	...	...
A-4255-1*	4	38 000	808	2.4	2.6	0.0016	160	548	...
A-4256-1*	4	38 000	1587	1.6	1.6	0.0010	230	1090	...
A-3851-1*	6 to 7	38 000	428	2.3	3.2	0.0045	53	219	...
A-5841-1*	0	40 000	21	...	...	...	...	...	...
A-5844-1*	1	40 000	15	0.8	Nil	...	...	...	...
A-5843-1*	1	40 000	25	1.6	Nil	...	...	...	...
A-5848-1*	2	40 000	537	0.8	Nil	0.0019	275	...	...
A-5849-1*	2 to 3	40 000	793	1.6	0.9	0.0013	400	...	...
A-4276-1*[a]	3	40 000	124	1.5	1.6	0.005	...	...	...
A-4277-2*[a]	3 to 4	40 000	246	3.1	1.6	0.0045	54	216	...
A-4277-1*[a]	3 to 4	40 000	396	2.3	2.1	0.0026	61	325	...
A-5305-1*[a]	4	40 000	79	1.6	Nil	0.008	...	...	...
A-5307-2*[a]	4	40 000	120	1.6	0.8	0.010	54	...	...
A-5314-2*	4	40 000	196	1.6	0.8	0.0035	67	...	...
A-5314-1*	4	40 000	1022	3.3	2.3	0.002	60	428	...
A-4567-1*[b, c]	4	40 000	1764	1.6	1.0	0.0006	520	...	...
A-4569-1*[a]	4 to 5	40 000	445	2.4	Nil	0.002	87	...	...
A-4568-1*[b, d]	5	40 000	748	...	...	0.0009	350	...	...
A-4566-1*	7	40 000	1025	4.0	4.8	0.0027	70	285	...
A-4095-2*	5 to 6	45 000	150	2.3	3.2	0.012	23	78	...
Tested at 1800 F.									
A-5846-2**[e]	−2	25 000	2.1	...	...	...	...	...	...
A-5847-2**[e]	−2	25 000	2.6	3.1	2.4	...	...	...	...
A-5845-2**[e]	−1	25 000	1.8	1.6	...	...	...	...	...
A-5841-2*[e]	0	25 000	2.9	0.8	Nil	...	...	...	...
A-5844-2*[e]	1	25 000	21.6	3.3	Nil	...	...	...	...
A-5843-2*[e]	1	25 000	21.7	4.0	0.8	...	...	...	...
A-6079-1**	2 to 3	25 000	11.8	9.6	8.0	...	...	...	...
A-6079-2**	2 to 3	25 000	22.4	8.1	8.0	...	...	...	...
A-6086-2*	3	25 000	99.1	5.6	6.3	0.0195	45	72	...
A-6086-1*	3	25 000	132.4	4.1	4.0	0.0086	73	104	...
A-4257-2*	3 to 4	25 000	186	10.2	8.8	0.016	46	91	152
A-6078-2*	4	25 000	100.3	4.8	1.6	...	...	...	...
A-6078-1*	4	25 000	103.3	6.4	6.3	0.017	51	72	...
A-4569-2*[a]	4 to 5	25 000	53	1.6	1.2	...	...	...	...
A-6085-1*	4 to 5	25 000	88.7	6.4	4.0	0.024	30	52	...
A-6085-2*	4 to 5	25 000	108.9	5.6	4.8	0.019	36	71	...

* 58Cr - 15Fe - 25Mo - 2Ti type alloy.

** 60Cr - 15Fe - 25Mo type alloy.

[a] Alloy was melted at atmospheric pressure under a slag.

[b] Specimen was solution treated for 50 hr. at 2400 F. and then aged for 96 hr. at 1600 F.

[c] Adapter threads failed at 1315 hr. Specimen was reloaded with another adapter and tested to rupture.

[d] Adapter threads failed at 748 hr. Specimen was broken during reassembly.

[e] Casting cooled to room temperature in the mold.

NOTE 1.—Unless noted to the contrary, the alloys were melted in vacuum.

NOTE 2.—Unless noted to the contrary, the specimens were stress relieved at 1200 F.

MECHANICAL AND PHYSICAL PROPERTIES

Creep-Rupture Data:

Chromium-base alloys were tested in creep rupture at either 1600 or 1800 F. To minimize premature breaking of the bars during application of the load, the following procedure was generally used: Each specimen was heated to temperature in the test unit, loaded to about two-thirds of the test stress for approximately 1 hr., and then loaded to the test stress.

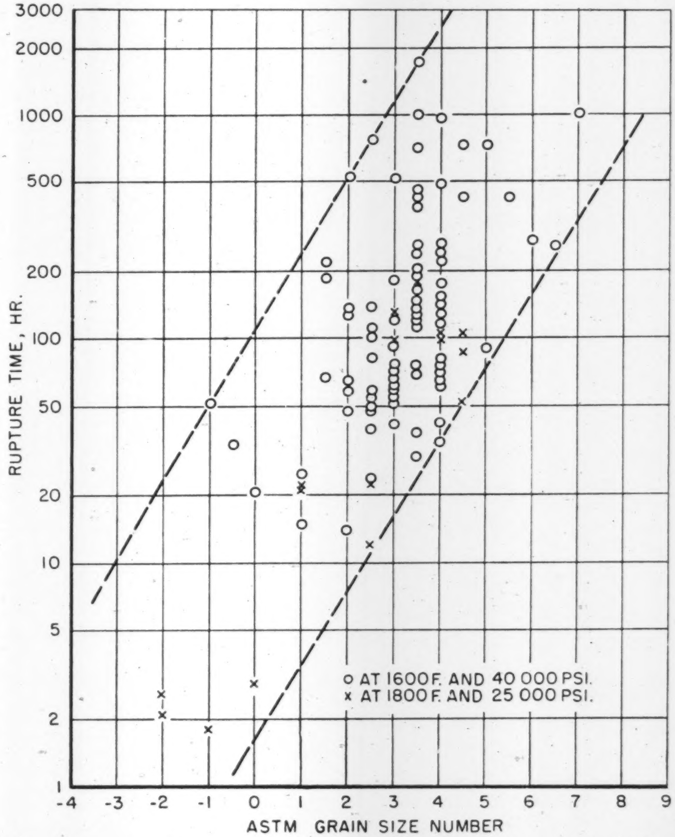


FIG. 8.—Grain Size *versus* Rupture Time for Chromium-Base Alloys at 1600 and 1800 F.

The creep-rupture properties for some of the alloys tested at 1600 and 1800 F. are shown in Table II and Fig. 7. The properties for a cobalt-base alloy, designated as X-40 or Stellite No. 31, are also shown in Fig. 7.

All of the alloys were tested at 1600 F. in the cast and stress-relieved condition, except for Heats A-4567 and A-4568. These two heats were solution treated for 50 hr. in purified argon at 2500 F., air-cooled to room temperature, aged for 96 hr. in purified argon at 1600 F., and furnace-cooled to room temperature. The 1800 F. tests were made on alloys either as-cast or cast and stress-relieved.

As illustrated in Fig. 7, the 100-hr. rupture strengths of the best 60Cr - 15Fe - 25Mo and 58Cr - 15Fe - 25Mo - 2Ti type alloys at 1600 F. are 44,000 and 55,000 psi., respectively. These values are superior to the 1600 F. rupture strength for Stellite No. 31, one of the best cobalt-base alloys (21,000 psi.).

The addition of titanium to the 60Cr - 15Fe - 25Mo type alloy markedly refined the grain structure and improved the rupture strength. The 22 bars which showed the longest rupture times at 1600 F. contained 1.63 to 2.13 per cent titanium. The strongest bar (A - 4567 - 1) gave a rupture time of 1764 hr. at 1600 F. and 40,000 psi.; this contained 2.01 per cent titanium and was in the solution-treated and aged condition.

TABLE III.—BEND-TEST RESULTS FOR CHROMIUM-BASE ALLOYS.

Heat Number	Remarks	A.S.T.M. Grain Size Number[a]		Deflection, in.[b]		Maximum Bending Stress, psi.[b]	
		Max.	Min.	Max.	Min.	Max.	Min.
	Effect of Carbon Content						
A-5840**	0.01% C	5	5	0.0063	0.0050	126 000	92 100
A-5820*	0.03% C	4 to 5	2	0.0035	0.0017	60 400	45 700
A-5818*	0.05% C	3	1	0.0025	0.0015	50 400	34 900
A-5819*	0.06% C	3	2	0.0035	0.0020	63 200	42 000
A-5811*	0.16% C	5	4 to 5	0.0038	0.0035	73 500	58 700
	Effect of Mold Temperature						
A-5811*	Mold Temperature = 1200 F.	5	4 to 5	0.0038	0.0035	73 500	58 700
A-5821*	Mold Temperature = 900 F.	4	3	0.0036	0.0022	65 800	50 000
A-5822*	Mold Temperature = 600 F.	4 to 5	2 to 3	0.0037	0.0027	68 500	50 200
A-5823*	Mold Temperature = 300 F.	5 to 6	5	0.0051	0.0033	70 800	63 300
	Effect of Alloy Additions						
A-6089**	0.04% B	5	4	0.0068	0.0054	100 500	87 000
A-6090**	0.1% B	4	3 to 4	0.0065	0.0057	106 800	81 800
A-6091**	0.5% B	5	4	0.0059	0.0047	101 200	73 500
A-6092**	2.8% B	...	...	0.0024	0.0016	40 400	31 600
A-6087**	0.003% N	4 to 5	3	0.0054	0.0040	87 400	66 100
A-6088**	0.005% N	4	2	0.0048	0.0040	84 100	58 500
A-6093**[c]	0.36% N	0	0	0.0043	0.0024	57 800	41 400
A-6094**[c]	0.86% N	0	0	0.0031	0.0024	44 100	38 900
A-6095**[c]	1.48% N	...	...	0.0043	0.0027	70 300	47 100
A-6096**[c]	Nil Ag	3 to 4	3	0.0055	0.0045	87 800	71 700
A-6099**[c]	0.04% Ta	4 to 5	3	0.0040	0.0032	64 500	54 800
A-6400**	0.08% Be	1 to 2	0	0.0043	0.0022	65 200	35 500
A-6402**	0.06% W	5	3	0.0079	0.0058	122 800	88 000
A-6403**	0.54% W	4	3	0.0055	0.0025	88 000	65 600

* 58Cr - 15Fe - 25Mo - 2Ti type alloy.

** 60Cr - 15Fe - 25Mo type alloy.

[a] The maximum and minimum A.S.T.M. grain size numbers are for individual bars from the same heat. They do not represent a range of grain sizes for any one bar from the heat.

[b] The maximum and minimum values for deflection and bending stress were not necessarily on bars having the maximum and minimum grain size numbers.

[c] Alloy melted at atmospheric pressure. All other alloys were melted in vacuum.

NOTE.—The castings were tested in the as-cast condition.

In general, the ductility of the 60Cr - 15Fe - 25Mo type alloy was low. In the rupture tests at 1600 F., the elongation values for this alloy were approximately 1 to 5 per cent, as compared with 5 to 14 per cent for the 60Cr - 25Fe - 15Mo type alloy. The ductility values for the latter material compare favorably with those for one of the strongest cobalt-base alloys (designated as Stellite No. 31 or X-40).

Seven bars which had been tested at 1600 F. and 38,000 to 45,000 psi. for various periods of time were analyzed for nitrogen. It was found that a bar which originally contained 0.05 per cent nitrogen picked up 0.07 per cent nitrogen in 38 hr. and 0.24 per cent in 1590 hr. The relationship between nitrogen pickup and time of test was linear.

The addition of approximately 2 per cent titanium to the 60Cr - 15Fe - 25Mo type alloy not only increased the rupture time at 1600 F. and 40,000 psi., but decreased the minimum creep rate (Fig. 7).

Sixteen bars were tested in creep rupture at 1800 F. and 25,000 psi. (Table II). Some of these bars showed outstanding rupture times of 100 hr. or more at 25,000 psi.

The creep-rupture data obtained at 1600 and 1800 F. were examined to ascertain the reasons for the variations in the rupture times of bars having similar analyses. The only variable that showed an effect on rupture life was grain size. The rupture time generally increased with decreasing grain size (Fig. 8).

Metallographic examination of the rupture bars, after testing at 1600 or 1800 F., revealed transcrystalline fractures. The type of fracture will be discussed in more detail later.

Bend-Test Data:

Since chromium-base alloy bars broken at room temperature, 1600 F., and 1800 F. showed transcrystalline fractures, the equicohesive temperature for this material appears to be above 1800 F. Therefore, an improvement in the ductility of chromium-base alloys at room temperature should be reflected in an improvement in ductility at 1600 and 1800 F. Accordingly, the factors which influence the ductility at room temperature were investigated using room-temperature bend tests.

Seventy-eight chromium-base alloy heats were prepared for this study. The bend-test results for a few of these heats are recorded in Table III.

The maximum and minimum values for maximum bending stress (in tension), deflections, and A.S.T.M. grain size numbers are shown in Table III for five or six bars from each of several heats. Maximum bending stress was calculated from the breaking load by using the following relationship:

$$S = \frac{3WL}{2bd^2}$$

where:

S = maximum bending stress (in tension) in the outer fiber at time of fracture, psi.,
W = breaking load, lb.,
L = distance between knife-edge supports (2 in.).
b = specimen width (approximately 0.20 in.), and
d = specimen thickness (approximately 0.32 in.).

The values for deflection were obtained with a comparator micrometer mounted on the Amsler testing machine. The recorded values were measured at the 50-lb. load increment closest to the load at failure.

An analysis of all the data showed that only two of the variables, grain size and oxygen content, appeared to be related to maximum bending stress. These relationships are illustrated in Fig. 9.

The maximum bending stress tended to vary inversely as the logarithm of the grain size or directly as the A.S.T.M. grain size number. The maximum and minimum grain sizes given in Table III are those of individual bars from any given heat and do not represent a range of grain sizes in any one bar. Grain sizes were determined by examining the bars metallographically and comparing their grain sizes with standard A.S.T.M.

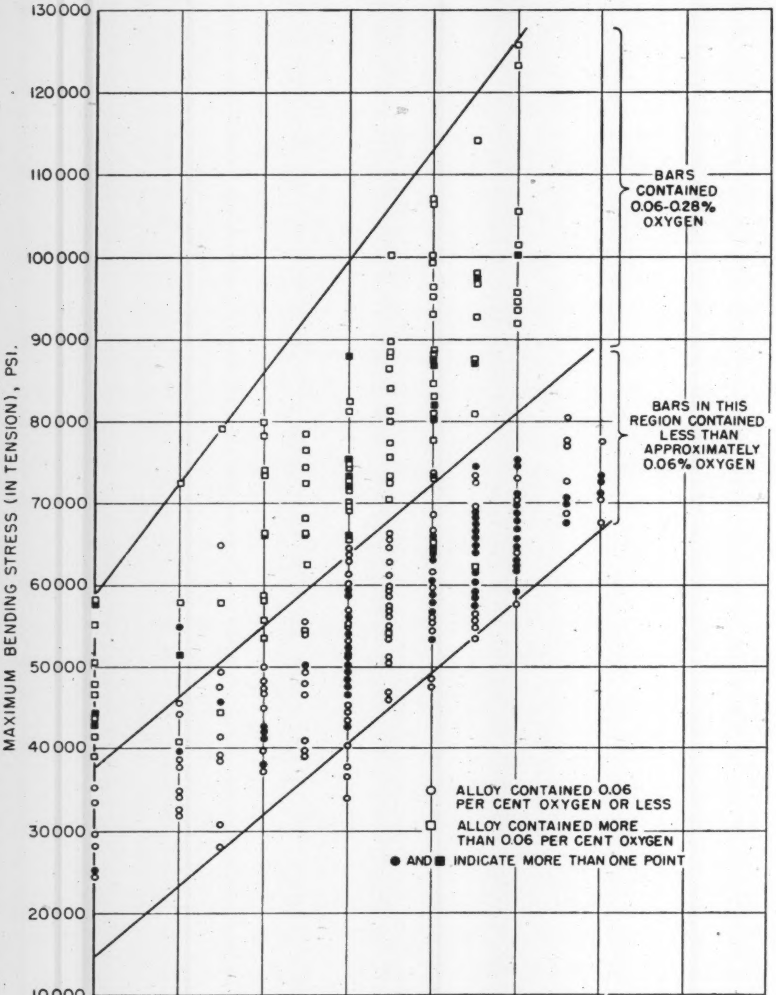


FIG. 9.—Grain Size *versus* Maximum Bending Stress (in Tension) at Room Temperature for Chromium-Base Alloys.

grain-size charts. Large and small grains did not occur in the same sample. The estimated grain size sometimes appeared to lie between two adjacent A.S.T.M. numbers. When this occurred, the sizes were shown as a range, for example, 3 to 4. For the sake of simplicity, in Fig. 9, points for such grain-size ratings were plotted midway between the two adjacent grain size numbers.

For any particular grain size, the maximum bending stress tended to increase with an increase in the oxygen content of the alloy. An exact relationship between the two was not established. The oxygen content which seemed to separate the data into the two zones shown in Fig. 9 is approximately 0.06 per cent. It is difficult to visualize how a high residual oxygen content in itself produced an increase in shock resistance. A high oxygen content (0.06 to 0.28 per cent) may simply have insured a low carbon content in the alloy. When the amount of carbon exceeds the limit of solubility, the carbide particles may precipitate microscopically on slip planes and impede deformation.

The room-temperature ductility for the best chromium-base alloy tested to date was compared with the values for one of the less ductile ferrous alloys, cast iron, and one of the least ductile of the cobalt-base alloys, Stellite No. 21 aged at 1350 F. for 50 hr. The load-deflection curve for Stellite No. 21 showed that considerable plastic deformation occurred during testing; the curve for cast iron was nearly linear, indicating only a small amount of plastic deformation. With one exception, which will be discussed later, the plots for all of the chromium-base alloys tested were straight lines, which indicated that the specimens had deformed elastically but not plastically. The area under the curve for the chromium-base alloy was approximately the same as that for the cast iron. From this observation, it appears that the work required to break chromium-base alloys is of the same order of magnitude as that

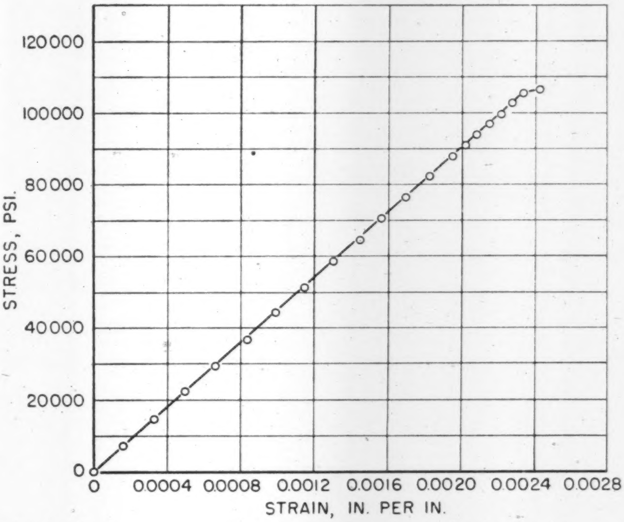


FIG. 10.—Stress-Strain Curve for 60Cr - 15Fe - 25Mo - 0.06W Alloy in Bending at Room Temperature.

to break cast iron. The area under the curve for aged Stellite No. 21 was about 40 times that for the chromium-base alloy.

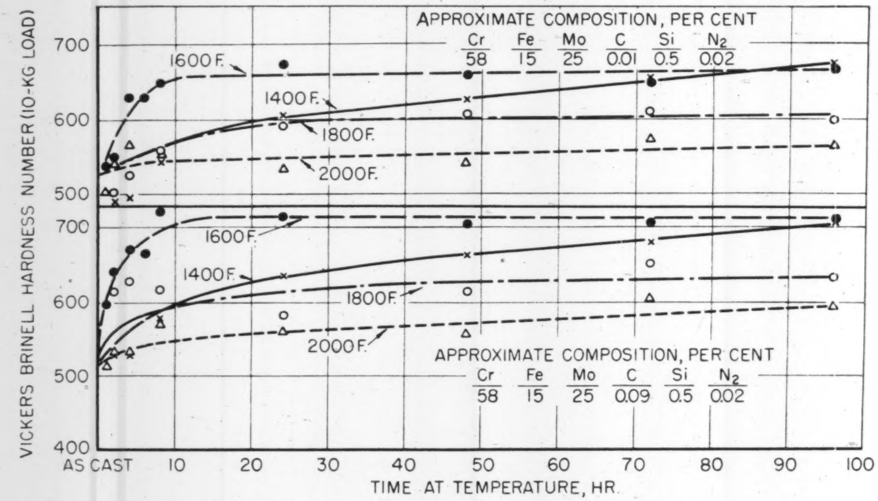


FIG. 11.—Aging Curves for 60Cr - 15Fe - 25Mo Type Alloy.

Nine chromium-base alloy bars were loaded to point of fracture with SR-4 strain gages attached to the side stressed in tension. The use of SR-4 strain gages permits measuring small amounts of plastic deformation that would not be detected with a comparator micrometer. Only one bar (from Heat A-6402) showed evidence of plastic deformation; the stress-strain curve is shown in Fig. 10. The amount of plastic deformation is, however, very small. Metallographic examination of this bar after testing revealed the presence of mechanical twins, indicating plastic deformation. This subject is discussed more fully later.

Impact Data:

To evaluate the relative toughness and to determine whether a transition temperature existed between 1800 F. and room temperature, unnotched Charpy bars of the 58Cr - 15Fe - 25Mo - 2Ti type alloy, as cast, were tested in impact at five temperatures. The bars were heated to slightly above the test temperature, held for 30 min., and tested. The energies required to break the bars at the various temperatures were:

Temperature, deg. Fahr.	Energy, ft-lb.
80	½
800	1
1200	1
1600	¼
1800	1½

These data verified bend and creep-rupture test results in that (1) the 58Cr - 15Fe - 25Mo - 2Ti type alloy is shock sensitive at room temperature as well as at 1800 F., and (2) the alloy does not have a transition temperature below 1800 F.

Examination of two specimens of the 60Cr - 15Fe - 25Mo type alloy by the X-ray diffraction method showed room-temperature cleavage on the (100) plane.

Hardness Data:

The effect of aging on the hardness of chromium-base alloys was investigated. Specimens were heated in air to the desired temperature, held at temperature for various periods of time, and then cooled to room temperature. An average Vickers hardness value (10-kg. load) was determined for each specimen. Figure 11 summarizes the hardness-aging time data.

To obtain a sufficient number of specimens for this hardness survey, it was necessary to use all available material from the castings. Hardness readings taken on specimens from the central portion of the casting may have been low because of unsoundness in that section of the casting. In a survey of six specimens held at temperature for 96 hr., the hardness across the specimens varied more than 100 Vickers numbers. The curves, therefore, are only indications of the trends of the effect of aging at various temperatures.

A solution-treated specimen from the 60Cr - 15Fe - 25Mo type alloy had a hardness value of 480 Vickers. The microstructure of this sample consisted solely of chromium-rich solid solution (Fig. 5(*d*)). At this high level of hardness, room-temperature ductility would be expected to be low, but not necessarily practically zero. High-purity chromium (0.014 per cent O_2, 0.009 per cent C, and 0.002 per cent N_2) produced by the iodide decomposition process, on the other hand, has a Vickers hardness value of approximately 100. This chromium metal is hot ductile, and at low strain rates is cold ductile. As more is learned about the factors affecting the ductility of chromium, the chances of producing more ductile chromium-base alloys become greater.

Oxidation Data:

Bars of the 60Cr - 15Fe - 25Mo type alloy generally acquired thin adherent oxidation films during rupture testing at 1600 and 1800 F. A few bars revealed severe localized oxidation. To throw some light on the high-temperature scaling characteristics of the alloy, oxidation-corrosion tests were made under the following conditions:

1. Continuous and intermittent exposure at 1600 and 1800 F. for 100 hr. in air saturated with water at 90 F.
2. Continuous exposure at 1600, 1800, and 2000 F. for 100 hr. in a *low-oxygen* atmosphere (99 per cent nitrogen, 1 per cent oxygen) saturated with water at 90 F.
3. Continuous exposure of *preoxidized* specimens at 1600 and 1800 F. in air saturated with water at 90 F. Preoxidation was accomplished by heating at 1800 F. for 25 hr. in air saturated at 90 F. with water.

The testing procedure followed that used for iron-nickel-chromium heat-resistant alloys by Brasunas, Gow, and Harder.[10]

In these tests, the appropriate atmosphere was saturated with water at 90 F., preheated so as not to cool the specimens during testing, and then passed through the furnace at a rate of 100 cu. cm. per minute. Specimens exposed intermittently to the test atmosphere were cooled to room temperature once each day, wiped to remove loose scale, weighed, and replaced in the furnace maintained at the test temperature. At the conclusion of the test period, the specimens were cathodically descaled in a molten caustic salt bath at 800 F. and water quenched. For descaling, a current of 400 amp. was used for 4 to 8 min., depending upon the adherence of the scale. After washing and drying, the specimens were reweighed to determine the amount of metal lost by corrosion. From the weight-change data and the surface area, the rate of metal loss was calculated in terms of grams per square inch per day and inches per year penetration.

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[10] A. D. Brasunas, J. T. Gow, and O. E. Harder, "Resistance of Iron-Nickel-Chromium Alloys to Corrosion in Air at 1600 to 2200°F.," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 870 (1946).

The results of all of the oxidation tests are illustrated in Fig. 12.

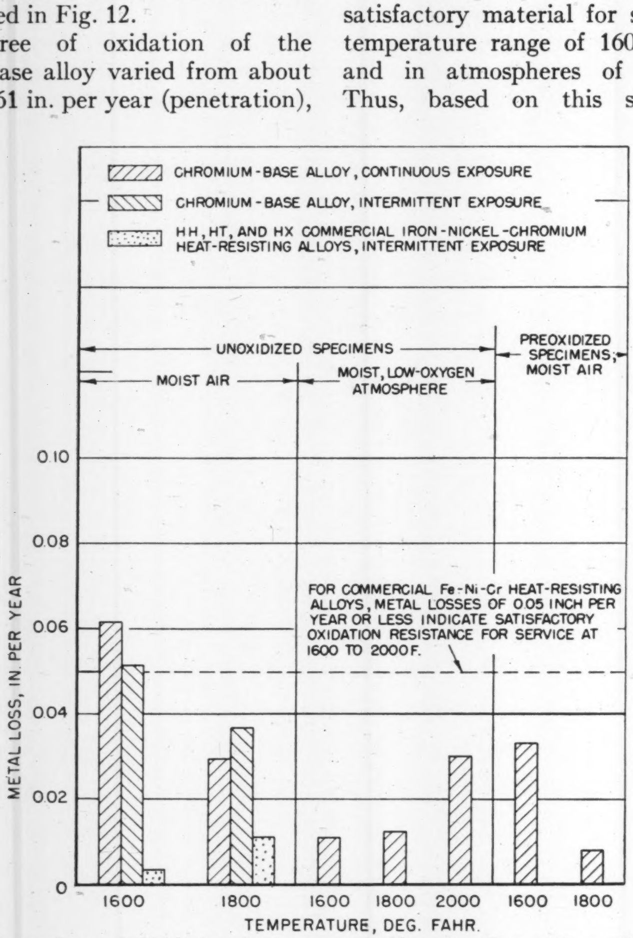

FIG. 12.—Comparison of Oxidation Resistance of Chromium-Base Alloy With Commercial Iron-Nickel-Chromium Alloys.

The degree of oxidation of the chromium-base alloy varied from about 0.008 to 0.061 in. per year (penetration), depending upon the exposure temperature, type of atmosphere, and condition of the specimen surface prior to exposure. The results in normal air showed that at 1600 F. the corrosion rate for this alloy was five to six times as great as that of the commercial iron-nickel-chromium alloys, and at 1800 F., three to four times as great. However, it should be pointed out that for commercial Fe - Ni - Cr heat-resisting alloys an oxidation rate of 0.05 in. per year or less indicates a satisfactory material for service in the temperature range of 1600 to 2000 F. and in atmospheres of these types. Thus, based on this standard, the 60Cr - 15Fe - 25Mo type alloy merits a good oxidation-resistance rating.

These tests show that for unknown reasons the 60Cr - 15Fe - 25Mo type alloy corrodes more readily in normal air at 1600 F. than at 1800 F., even when the alloy is prescaled prior to oxidation testing. The reason for the severe localized corrosion of occasional bars during creep-rupture testing is not obvious. Possibly this localized corrosion results from the accelerating action of molybdenum oxide (MoO_3) as an oxidation catalyst at the surfaces of the specimens, as described by Leslie and Fontana.[11]

Other forms of surface instability, such as intergranular penetration or decarburization, were not observed in any of the specimens tested.

Thermal-Shock Data:

The resistance to thermal shock of the chromium-base alloy was evaluated under conditions simulating stator-blade service. Aged Stellite No. 21 specimens were used for comparison standards. Six wedge-shaped specimens from each material were tested. Individual specimens were heated for 15 min. at 1600 or 1800 F., held in an air blast for 5 min., and then examined for cracks by Zyglo inspection. Air was delivered at 70 F. from a pressure tank down through a rectangular orifice at a velocity of 130 ft. per sec. and at a constant pressure of 50 psi. Baffles were placed in the orifice chamber to provide uniform flow conditions at the exit. The specimens were supported so that the thin top edge of the specimen was $\frac{5}{8}$ in. below the orifice.

Three specimens of each of the alloys showed no cracks after 25 cycles of rapid cooling from 1600 F. In tests using other specimens and temperature of 1800 F., two of the three specimens of the chromium-base alloy cracked on the 49th cycle; the remaining specimen had not cracked after 63 cycles. The three specimens of aged Stellite No. 21 did not crack after 65 cycles from 1800 F. For a material showing practically no room-temperature ductility, the resistance to cracking of the chromium-base alloys in this thermal-shock test was very encouraging.

DISCUSSION ON MICROCRACKS

There has been considerable interest in the role of microcracks in brittle material.[12, 13, 14] The following discussion is presented to show that this type of defect may have been present in the chromium-base alloys tested.

Griffith[14] suggested that the tensile strength of actual solids is lower than that of an ideal crystal because of microcracks and resulting stress concentrations. He assumed that a crack will propagate only if, for a small increase of length, the work of external forces will balance both the decrease in elastic energy around the crack and the increase in surface energy. On this basis, the stress required to propagate a crack (an elliptical hole of major axis equal to $2C$) is

$$S = \sqrt{\frac{2\alpha E}{\pi C}} \quad \ldots\ldots\ldots (1)$$

or

$$S = K\sqrt{\frac{\alpha E}{C}} \quad \ldots\ldots\ldots (2)$$

where:

α = energy per unit area to form a new surface,

E = modulus of elasticity,

C = one-half the length of the crack, and

K = constant which depends on the shape of the crack.

For polycrystalline aggregates, the condition for a crack to propagate across a grain boundary may be the attainment of a critical stress at the end of the crack. Orowan[13] stated that, "since the stress at a given distance from the tip of the crack is proportional to the square root of its length, one would expect the brittle strength of a polycrystalline metal to be inversely proportional to the square root of the mean grain diameter. This was confirmed in the case of polycrystalline zinc, and recently the same relationship was found between the grain size and the brittle strength in several steels; the brittle strength was measured by fracture in liquid nitrogen." This relationship is shown as follows:

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[11] W. C. Leslie and M. G. Fontana, "Mechanism of the Rapid Oxidation of High-Temperature, High-Strength Alloys Containing Molybdenum," *Transactions*, Am. Soc. Metals, Vol. 41, p. 1213 (1949).

[12] Clarence Zener, "Elasticity and Anelasticity of Metals," The University of Chicago Press, p. 129–131 (1948); and "The Micro-Mechanism of Fracture," Fracturing of Metals, Am. Soc. Metals, pp. 3–31 (1948).

[13] E. Orowan, "Fracture and Strength of Solids," Unclassified Atomic Energy Publication, *NP-1077*, Ministry of Supply, United Kingdom (1949).

[14] A. A. Griffith, "The Phenomena of Rupture and Flow in Solids," *Philosophical Transactions*, Royal Society (London) p. 163 (1920); and "Theory of Rupture," *Proceedings*, First International Congress Applied Mechanics, Delft (1924).

$$S_e = \frac{K_1}{\sqrt{D}} \quad \text{.........} \quad (3)$$

where:

S_e = experimental fracture stress,

K_1 = constant, and

D = mean grain diameter.

Since the mean grain diameter varies inversely as the square root of the number of grains per unit of area N, Eq. 3 can be rewritten as follows:

$$S_e = K_1\sqrt[4]{N} \quad \text{........} \quad (4)$$

The data shown in Fig. 9 were replotted in Fig. 13 on log-log scales, and the results are in agreement with Eq. 4. The individual points in Fig. 13 are the average values of maximum bending stress for a constant grain size. The slope for the two lines shown in Fig. 13, for alloys containing low and high oxygen contents, is 0.22. The slope of the line given by Eq. 4 is 0.25. The relationship between microcracks and the slopes of the lines in Fig. 13 indicates that the length of the crack and the diameter of grains are the same in chromium-base alloys. On this basis, $\frac{1}{\sqrt{N}}$ can be substituted for the term C in Eq. 2. The value of stress at the time of fracture is indicated by

$$S = K\sqrt{\alpha E}\,\sqrt[4]{N} \quad \text{.....} \quad (5)$$

Figure 13 shows that two values of stress can be obtained with one value of

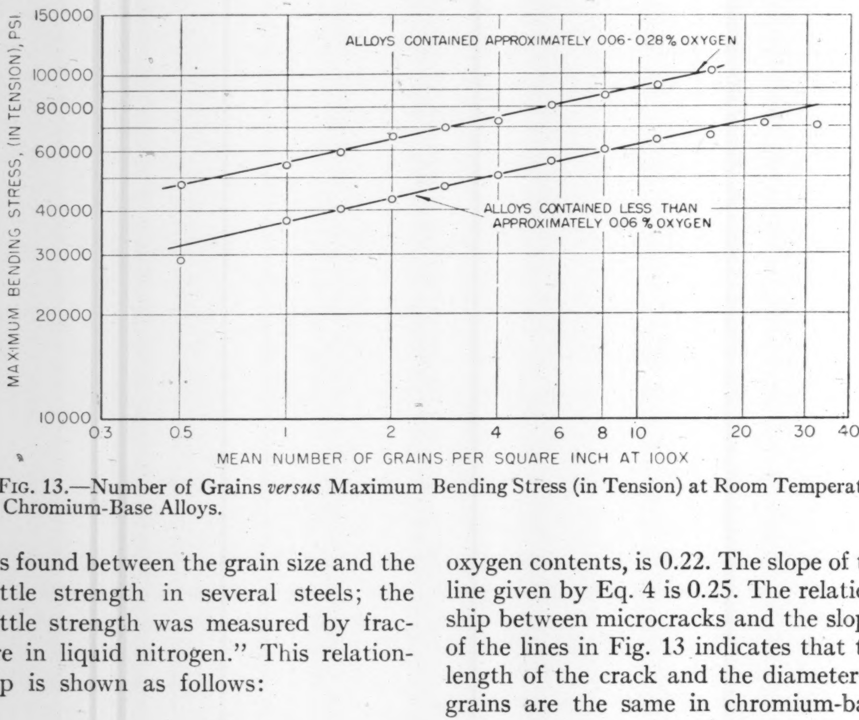


FIG. 13.—Number of Grains *versus* Maximum Bending Stress (in Tension) at Room Temperature for Chromium-Base Alloys.

N. The alloys with high oxygen contents had higher values of *S* than did the alloys with low oxygen contents. This condition could be explained by associating higher values of α with alloys having the higher oxygen contents. The speculation as to the possible influence of oxygen on surface energy and the presence of microcracks in chromium-base alloys may be of interest to future investigators.

DISCUSSION ON PLASTIC DEFORMATION

With one exception (Fig. 10), none of the stress-strain or load-deflection curves showed signs of plastic deformation in bending. However, there was other evidence of plastic deformation in chromium-base alloys. Bent-test bars showed slip lines and mechanical twins in areas adjacent to the fractures; twin bands were observed adjacent to Vickers hardness indentations (Fig. 14). After fracture, slip lines were visible on the test bar surfaces that were stressed in tension; this observation was facilitated by polishing these surfaces prior to testing. Slip lines were found in an alloy which contained 0.10 per cent carbon and 0.012 per cent oxygen; they were noted in areas of the microstructure where the chromium-rich solid-solution matrix phase predominated (Fig. 14 (*b*)). Twins were observed after fracture on the tension and compression sides of bend-test bars examined after testing (Fig. 14 (*a*)) and near hardness indentations (Fig. 14 (*c*)). These bands were found only in those alloys which had high oxygen contents, approximately 0.14 to 0.27 per cent. Twins were not noted in areas where carbides were present (Fig. 14 (*d*)). Twins and slip lines were not visible in the same specimen. These evidences of plastic deformation give some hope of developing ductility in chromium-base alloys.

Chromium-base alloy bars broken at room temperature, 1600 F., and 1800 F. showed transcrystalline fractures. This type of fracture resulted even when a carbide network surrounded the grains. Therefore, the lack of ductility appears to result from the brittleness of the chromium-rich solid-solution matrix phase.

The matrix phase in chromium-base alloys crystallizes with a body-centered-cubic structure. Chromium, iron, and molybdenum, the major components in these alloys, are body-centered-cubic transition elements. Tungsten and molybdenum have been considered ductile metals only since they have been produced in very pure form and worked by special procedures. There is no fundamental reason for the lack of ductility of chromium. When metal of sufficient purity is produced, chromium, too, may be cold ductile.

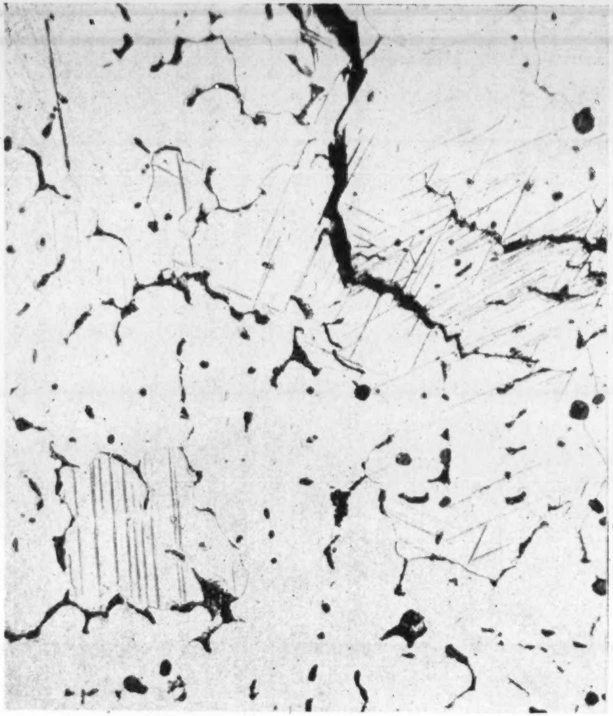
Lack of ductility in chromium-base alloys may be associated in part with the precipitation of some constituent. It is conceivable that carbides or nitrides might precipitate microscopically on slip planes. Slip is difficult in a strained lattice; the metal is hard and strong, and ductility is greatly reduced.

CONCLUSIONS AND SUMMARY

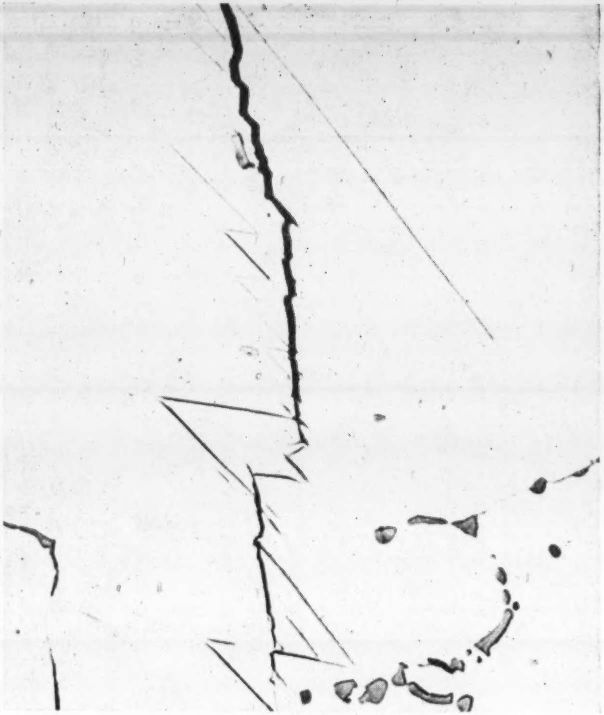
1. Most of the alloys studied in this investigation were prepared in the vacuum-melting and casting apparatus. A sufficient number of alloys were melted at atmospheric pressure under a protective slag to indicate that the air-melted alloys yielded mechanical properties which were almost as good as those of the vacuum-melted alloys.

2. The 60Cr-15Fe-25Mo type alloy is susceptible to the formation of grinding cracks and must be ground with care.

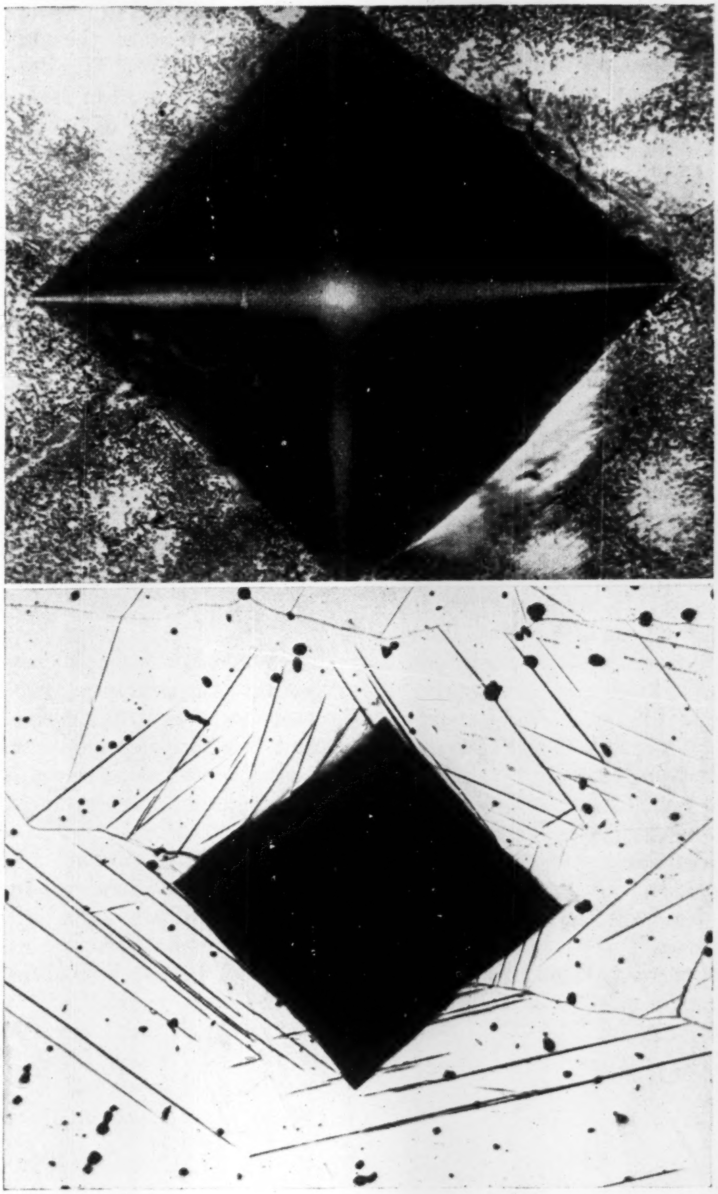
3. The effect of aging on the hardness of chromium-base alloys was investigated. Maximum hardness was obtained after 20 hr. at 1600 F. or after 100 hr. at 1400 F.



(*a*) Mechanical Twins in Fractured Bend-Test Specimen. Alloy Contained 0.01 per cent C and 0.14 per cent O_2. (× 250).



(*b*) Slip Lines in Fractured Bend-Test Specimen. Alloy Contained 0.10 per cent C and 0.012 per cent O_2. (× 1000).



(*c*) Mechanical Twins Adjacent to Vickers Hardness Indentation. Alloy Contained 0.01 per cent C and Approximately 0.14 per cent O_2. (× 250).

(*d*) Vickers Hardness Indentation. Alloy Containing 0.09 per cent C and Approximately 0.02 per cent O_2. Note Absence of Twins (× 500).

FIG. 14.—Slip Lines and Mechanical Twins in 60Cr - 15Fe - 25Mo Type Alloy.

4. The 60Cr - 15Fe - 25Mo type alloy merits a good oxidation-resistance rating on the basis of tests conducted at 1600, 1800, and 2000 F. in moist air and moist, low-oxygen atmospheres.

5. Several chromium-base alloy specimens had rupture times in excess of 1000 hr. at 1600 F. and 40,000 psi., and in excess of 100 hr. at 1800 F. and 25,000 psi.; these values are superior to the 1600 and 1800 F. rupture times for Stellite No. 31, one of the best cobalt-base alloys. The rupture times for chromium-base alloys at 1600 and 1800 F. increased with decreasing grain size.

6. Chromium-base alloys are sensitive to mechanical shock at room temperature as well as at temperatures up to 1800 F. They show transcrystalline fractures when broken at room temperature, 1600, or 1800 F. The presence or the location of an excess phase in the microstructure did not alter the type of fracture.

7. Thermal-shock tests were conducted on chromium-base alloy and aged Stellite No. 21 specimens by cooling rapidly from 1600 and 1800 F. Neither type of alloy failed after 25 cycles from 1600 F. The aged Stellite No. 21 specimens withstood 65 cycles from 1800 F. Two chromium-base alloy specimens failed after 49 cycles from 1800 F.; the third specimen withstood 63 cycles without cracking.

8. The room-temperature maximum bending stress (in tension) of chromium-base alloys was found to be proportional to the fourth root of the number of grains per unit of area. This observation lends additional support to the findings of Orowan[13] and Griffith[14] as to the presence and effect of microcracks in brittle materials.

For a constant grain size, the bending stress was higher for the alloys with oxygen contents of between 0.06 to 0.28 per cent than for those lower in oxygen.

9. Evidence of a limited amount of plastic deformation was found in chromium-base alloys; slip lines and mechanical twins were observed in fractured bend-test bars. In one test, the stress-strain curve showed evidence of plastic deformation. Slip lines were observed in alloys with high carbon contents, while twins were found in alloys relatively low in carbon.

Acknowledgment:

The authors wish to express their appreciation to the sponsoring agency, the Materials Section of the Office of Naval Research, U. S. Navy Department, for permission to publish these results.

It is a pleasure to acknowledge the help of the staff members of Battelle Memorial Inst. in preparing the test materials and in conducting some of the tests. The authors are particularly grateful for the contributions and assistance of Robert M. Parke, A. B. Westerman, and C. H. Lorig.

DISCUSSION

MR. J. D. NISBET[1] (*presented in written form*).—The authors of this paper are congratulated for their pioneering effort in this new and difficult field of developing chromium-base alloys. There are several points that I should like to make and a few questions that I should like to ask concerning this report.

We have reached the conclusion in our vacuum-melting activities at General Electric that alloys containing high quantities of an active element such as chromium cannot be satisfactorily air melted. The rough division in percentage of chromium which can be handled in air has been set at 30 per cent. Chromium, we think, can be handled satisfactorily if atmospheric contamination is eliminated up to a percentage of 60 per cent. Beyond 60 per cent, it is our opinion that both the atmosphere and the crucible reaction must be eliminated. Therefore, when melting alloys containing from 60 to 100 per cent chromium, it is necessary to have inert atmosphere arc-melting methods in a non-reactive, water-cooled copper crucible. The limits we have imposed upon titanium, for comparison with another very reactive element, are 1 per cent maximum in air, 15 per cent maximum in vacuum, and of course, up to 100 per cent in the inert atmosphere arc-melting technique.

The chromium-base alloys studied by the authors of this paper were extremely brittle regardless of whether they were air melted or vacuum melted. It would seem reasonable to expect that a better direction for studying the problem of brittleness in these materials would be to consider higher degrees of purity rather than the lower purity to be expected from air melting. There is a second important point which makes a comparison of air-melted and vacuum-melted materials difficult when the comparison is made on alloys as hard and brittle as the chromium-base alloys seem to be. Such materials already contain large quantities of brittle secondary phases; therefore, changes in the oxide and nitride content of such materials is difficult if not impractical to measure by a study of rupture strength. In contrast with this, the easiest way to study differences between air and vacuum-melted reactive alloys is to study solutions which contain only one phase; and therefore, properties of such materials are easily changed by small additions of oxides and nitrides.

I wonder about the conclusion in the first paragraph under "Bend Test Data" which states that the equicohesive temperature of this material appears to be above 1800 F. Therefore, the authors expect a study of room-temperature shock resistance to be indicative of ductility at 1600 to 1800 F. This seems to be inconsistent with the fact that many materials such as molybdenum and chromium are brittle at room temperature whereas they become ductile at some temperature far below their recrystallization temperature. The transition from brittle to ductile in these materials is similar to the so-called impact-transition temperature in steels. This is based on the assumption that the so-called equicohesive temperature is closely related to the recrystallization temperature which is known to be higher for these materials. One would expect, therefore, brittle characteristics as well as ductile characteristics below the so-called equicohesive temperature. One would not expect the behavior to be the same at room temperature and at 1600 to 1800 F. for chromium-base alloys. It is also true that high low-temperature ductility does not assure ductile behavior at high temperatures. This fact is well known in the study of high temperature materials.

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[1] Metallurgical Div., General Electric Co., Schenectady, N. Y.

I should like to ask the following questions:

1. Why was silver considered as an alloying ingredient?
2. Do the authors have photomicrographs available which clarify the differences between the structures classified as *S*, *Y*, *and Z*?
3. Are photomicrographs available showing the transgranular fractures after rupture tests?
4. Do the authors expect alloys rich in chromium and molybdenum, both brittle metals in fairly high purity form, to ever be ductile?
5. Has any work been done at Battelle on the properties of the saturated solution matrix phase of the compositions studied without the multitude of other phases present? Is not this matrix brittle?

MR. W. D. FORGENG[2] (*presented in written form*).—The authors are to be complimented for the thoroughness of their experimental work and their excellent analysis of the data. We have been working for a number of years with chromium-base alloys including those of the type covered in this paper and are able to substantiate a number of the conclusions reached by the authors. We have also observed that the fracture of these materials both at room temperature and elevated temperatures is always transcrystalline, and the presence of minor amounts of an excess phase appears to be without effect on the path of failure.

The lack of ductility and sensitivity of the alloys to shock may be an inherent property of the chromium-rich solid solution and whether increased purity of the chromium used in making the alloys will result in improved properties is a matter for conjecture. While the brittleness of the alloys may be associated with a visible precipitation of carbides or nitrides, it may also be a manifestation of some pre-precipitation stage of these constituents or of the sigma phase. It is possible that the improved shock resistance of the higher oxygen alloys is associated with the removal of carbon or nitrogen atom groups from sites where they might impede slip in the matrix.

MR. C. T. GREENIDGE (*author's closure*).—We really do not know whether these alloys will be ductile when their purity is increased. Recent work at Battelle, however, has indicated that chromium of higher purity than that usually available shows some signs of ductility. We have compressed individual crystals and polycrystalline pieces of iodide chromium and found that they will deform as much as 40 per cent cold. This is the first concrete evidence we have had that purity affects the ductility of chromium.

Along the same line, we have recently made some tests on high-purity molybdenum. It also seems to be more ductile than molybdenum of lower purity.

I think if we could determine what causes embrittlement of chromium, perhaps we could explain why chromium-base alloys are not ductile. Chromium fractures in about the same manner as these alloys; therefore, we suspect that whatever makes chromium nonductile also makes these chromium-base alloys nonductile.

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[2] Union Carbide and Carbon Research Laboratories, Inc., Niagara Falls, N. Y.

The statement with reference to melting the alloys in air does not quite check our experience. We have been able, without difficulty, to melt in air, under proper slags, alloys with up to 50 per cent of chromium. One reason for melting some of the alloys described in this paper in air was to see whether they could be produced by conventional melting techniques. Vacuum equipment is not plentiful in the country, so if we should need these alloys in quantity, they might have to be made by melting in air. We wanted to find out whether this was feasible.

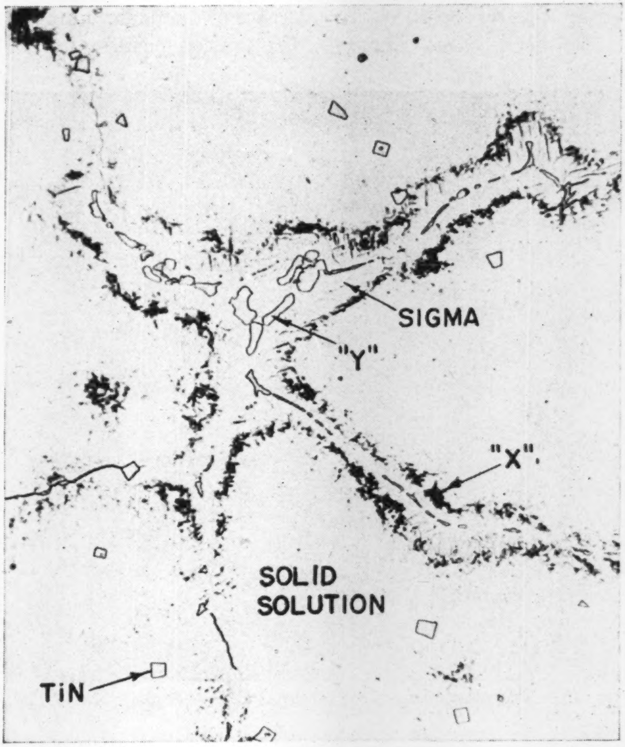

FIG. 15.—Microstructure of a 58Cr-20Fe-20Mo-2Ti Type Alloy in Cast and Stress-Relieved Condition (×500).

MR. W. L. HAVEKOTTE (*author's closure*).—The authors wish to take this opportunity to thank Messrs. Nisbet and Forgeng, for their discussion.

Silver, along with a number of other alloying elements, was added to the 60Cr-15Fe-25Mo type alloy in an attempt to refine the grain size. The other elements used were aluminum, boron, beryllium, carbon, titanium, vanadium, manganese, zirconium, tungsten, selenium, tantalum, and nitrogen. Since the alloy does not go through an allotropic change and could not be hot or cold worked to obtain grain refinement, a decrease in grain size could be obtained only by an alloy addition or by a change in solidification rate. Pouring and mold temperatures, mold designs, and mold compositions were varied to increase the rate of solidification, but these changes had no consistent effect on the grain size.

The microstructures shown in Fig. 5 illustrate the constituents in chromium-base alloys. Figure 15 is added to clarify the differences between these phases; this shows as-cast and precipitated carbides, sigma, and titanium nitride in a matrix of chromium-rich solid solution. Hammond[9] found the "Z" phase by X-ray diffraction methods, but did not identify it.

The 60Cr - 15Fe - 25Mo type alloy in its present state of purity and containing 0.08 per cent carbon was solution treated at 2500 F. for 50 hr. in purified argon. The microstructure of this alloy consisted of chromium-rich solid solution with a few oxide inclusions. However, even this specimen was brittle.

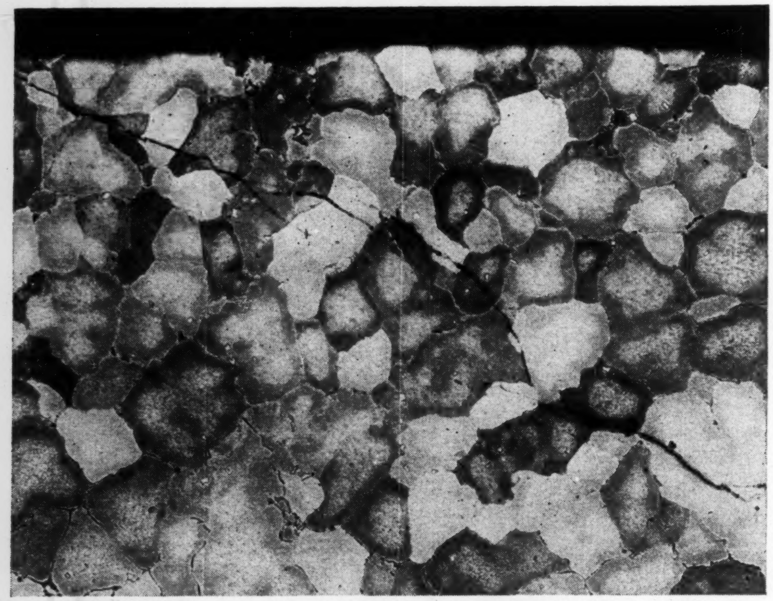
FIG. 16.—Microstructure of a Fractured Creep-Rupture Bar (×100).

Since the 60Cr - 15Fe - 25Mo type alloy is very brittle and showed transcrystalline fractures at room temperature, 1600 F, and 1800 F., an improvement in room-temperature ductility should be reflected in an improvement at 1600 and 1800 F. The bend-test data obtained at room temperature indicated that, as the grain size decreases, the maximum bending stress (in tension) gradually increases. Grain size seems to affect rupture time at 1600 and 1800 F. in a similar manner; rupture time tends to increase with decreasing grain size. Therefore, we believe that changes leading to an increase in ductility at room temperature will have a similar effect at these high temperatures.

Figure 16 shows the fractured surface and a crack just below this surface for a creep-rupture bar which withstood 38,000 psi. at 1600 F. for 1587 hr. It is evident that the specimen fractured in a transcrystalline manner.

SPRAY TESTING WITH NATURAL AND SYNTHETIC SEA WATER. PART I.—CORROSION CHARACTERISTICS IN THE TESTING OF METALS*

By Thomas P. May[1] and Allen L. Alexander[2]

Synopsis

Generalizations about the chemical analysis of ocean water are used as a basis for comparing several existing formulas for synthetic solutions which have served as substitutes for natural ocean water in certain laboratory test procedures. This comparison reveals certain faults in these formulas.

A new formula is proposed with exact reproduction of ion concentrations above 1 ppm. which are existent in natural ocean water. Provision is made for adjustment of pH and for the addition of heavy metal ions when desired.

For characterizing the corrosive properties of this solution, salt spray tests have been conducted upon a number of metals using as spray solutions natural sea water, synthetic sea water, 3 per cent and 20 per cent sodium chloride solutions. The same metals have been exposed in the atmosphere and in sea water at Kure Beach, N. C.

Zinc and zinc-plated steel corrode in spray tests using natural or synthetic sea water in a manner somewhat similar to their corrosion in natural marine environments. Such tests, however, are not necessarily "accelerated" since the corrosion rate in a marine environment may be higher than in the spray test. The corrosion of zinc and zinc-plated steel in spray tests with sodium chloride is not like the corrosion in the natural environments. Likewise, nickel-plated steel and bare steel do not suffer the same type of corrosion in spray tests as they do in a marine environment. Nickel-plated steel corrodes in a similar manner in the spray tests and in sea water but not in a marine atmosphere.

Several years ago at the Naval Research Laboratory it became desirable to conduct some corrosion experiments in synthetic sea water since at the time it was not practical to do the work in natural ocean water. A preliminary examination of existing formulas for synthetic sea water revealed serious discrepancies between them and no basis for eliminating these discrepancies was immediately apparent. Therefore, a literature survey was made to establish as nearly as possible what various analyses of ocean water have revealed (1).[3] This permitted the development of a formula which would provide an exact duplicate of the inorganic components of natural ocean water. During the last two years a number of corrosion tests have been run in order to evaluate this solution as a corrosive medium. Tests have been conducted in spray cabinets and in the natural environments at Kure Beach, North Carolina.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Corrosion Engineering Section, Development and Research Dept., The International Nickel Co., New York, N. Y.

[2] Protective Coatings Branch, Chemistry Division, Naval Research Laboratory, Washington, D. C.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1141.

Because of its interest in the spray test, Subcommittee III of A.S.T.M. Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys requested that the results of this study be presented as a matter of considerable interest to the Society. Section A on Corrosivity, Subcommittee VI on Methods of Testing of Committee D-19 on Industrial Water, also has a direct interest in this solution as a possible medium for tests.

Composition of Waters in Mid-Ocean

Natural ocean water is a solution of solids and gases which also contains detritus and live marine organisms, both plant and animal. The only important gases are oxygen and carbon dioxide, the concentrations of which are determined by the salinity, temperature, and the partial pressures of the gases in the atmosphere. In the usual types of corrosion experiments there is no simple way of controlling the concentrations of these dissolved gases. Neither has it proved practical to breed live marine organisms in solutions prepared in the laboratory. On the other hand the dissolved inorganic components other than gases may be readily duplicated in a synthetic solution.

A review of over one hundred and sixty sources, especially two (2, 3) reveals several generalizations which are valid for ocean waters in all of the major oceans:

1. The composition along seacoasts may vary considerably with location. River waters cause dilution and introduce local variations in the relative abundance of the ions. Rivers and harbors also may introduce pollution. Analyses of coastal waters therefore must be considered with caution.

2. The salt content in mid-ocean varies from one ocean to another, between different regions in a given ocean, and in a given region with the time of year. Wust (4) showed this change to be a linear function of the difference between evaporation and precipitation. The salinity also varies with depth because of stratified ocean currents.

3. A very important generalization is that, regardless of the absolute concentration, the relative proportions of the major components are virtually constant. This rule does not hold for silicate and nitrate ions which will vary in concentration depending upon the metabolism of marine organisms in different localities or in different seasons. It will also fail to hold for a number of trace elements.

TABLE I.—ELEMENTS IN NATURAL OCEAN WATER WITH CHLORINITY OF 19.00.

Element	ppm.	Element	ppm.
Chlorine	18980.	Iron	0.002 to 0.02
Sodium	10556.	Manganese	0.001 to 0.01
Magnesium	1272.	Copper	0.001 to 0.01
Sulfur[a]	884.	Zinc	0.005
Calcium	400.	Lead	0.004
Potassium	380.	Selenium	0.004
Bromine	65.	Cesium	0.002
Carbon[b]	27.5	Uranium	0.0015
Strontium	13.3	Molybdenum	0.0005
Boron[c]	4.6	Thorium	0.0005
Silicon[d]	0.02 to 4.0	Cerium	0.0004
Fluorine	1.3	Silver	0.0003
Nitrogen[e]	0.01 to 0.70	Vanadium	0.0003
Aluminum	0.5	Lanthanum	0.0003
Rubidium	0.2	Yttrium	0.0003
Lithium	0.1	Nickel	0.0001
Phosphorus	0.001 to 0.10	Scandium	0.00004
Barium	0.05	Mercury	0.00003
Iodine	0.05	Gold	0.000006
Arsenic	0.01 to 0.02	Radium	$2\text{–}3 \times 10^{-11}$

[a] Normally present as sulfate but may be present as sulfide in stagnant waters or in bottom sediments.

[b] Almost entirely bicarbonate ion when organic matter is discounted.

[c] Probably present in undissociated boric acid.

[d] Represents silicon present as silicate. Lower values noted in surface layers.

[e] Includes only nitrate, nitrite, and ammonia.

4. The pH of mid-ocean waters is usually in the range 8.1 to 8.3, depending upon salinity and the partial pressure of carbon dioxide in the air, but may vary between the limits of 7.5 and 8.4.

The concentration of sea water is

frequently expressed in terms of chlorinity rather than salinity since chlorinity is easier to determine by direct methods. Its numerical value is obtained from the amount of silver required to precipitate the halides in 328.5233 g. of ocean water. An average value for mid-ocean water is 19.381 g. of halides per kg. of ocean water.

The constancy of the proportions of the different ions is expressed in a term called the Cl-ratio which is the ratio of the concentration of an ion to the chlorinity.

Table I presents the concentrations in parts per million of elements in natural sea water with a chlorinity of 19.00. Table II contains the compositions of several synthetic solutions which supposedly duplicate the composition of natural ocean water. The formula in column two has been used to test the corrosion resistance of stainless steels (5); the one in column three for testing grease type rust preventive compounds (6). The formula of McClendon, Gault, and Mulholland (7) in the fourth column was developed to study certain marine organisms and contains components such as nitrates which are essential in the metabolism of the organisms.

For many purposes it is probable that many elements present in low concentration need not be present in a synthetic solution. On this basis Subow (8) and Lyman and Fleming (9) developed the formulas in columns five and six, respectively. Assuming that elements below approximately one part per million are not important in many applications, the formula in the last column of this table has been developed and, for situations where the less concentrated elements are desired, provision is made for adding them in the form of their nitrates.

The degree in which these solutions compare with natural ocean water is

TABLE II.—SUBSTITUTE SOLUTIONS FOR NATURAL OCEAN WATER.

Compound	Grams per Liter					
	46S18[a]	14-0-15[b]	M-G-M[c]	Subow[d]	L and F[e]	Proposed
NaCl	23.0	25.0	26.726	27.175	23.476	24.54
$MgCl_2 \cdot 6H_2O$	11.0	11.0	....	....	....	11.11
$MgCl_2$	....	....	2.260	2.507	4.981	....
$Na_2SO_4 \cdot 10H_2O$	8.0	....	....	....	....	....
Na_2SO_4	....	4.0	....	....	3.917	4.094
$CaCl_2 \cdot 6H_2O$	2.20	....	....	....	....	....
$CaCl_2$	....	1.20	1.153	1.170	1.102	1.159
KCl	0.20	....	0.721	0.743	0.664	0.695
KBr	0.90	....	....	....	0.096	0.101
$MgSO_4$	....	....	3.248	3.387	....	....
$NaHCO_3$	....	....	0.198	0.207	0.192	0.201
NaBr	....	....	0.058	0.085	....	....
H_3BO_3	....	....	0.058	....	0.026	0.027
Na_2SiO_3	....	....	0.0024	....	....	....
$Na_2Si_4O_9$	....	....	0.0015	....	....	....
H_3PO_4	....	....	0.0002	....	....	....
$AlCl_3$	....	....	0.013	....	....	....
NH_3	....	....	0.002	....	....	....
$LiNO_3$	....	....	0.0013	....	....	....
$SrCl_2$	....	....	....	....	0.024	....
NaF	....	....	....	....	0.003	0.003
$SrCl_2 \cdot 6H_2O$	....	....	....	....	....	0.042
$Ba(NO_3)_2$	....	....	....	....	....	0.994×10^{-4}
$Mn(NO_3)_2 \cdot 6H_2O$	....	....	....	....	....	0.546×10^{-4}
$Cu(NO_3)_2 \cdot 3H_2O$	....	....	....	....	....	0.396×10^{-4}
$Zn(NO_3)_2$	....	....	....	....	....	0.151×10^{-4}
$Pb(NO_3)_2$	....	....	....	....	....	0.066×10^{-4}
$AgNO_3$	....	....	....	....	....	0.0049×10^{-4}

[a] Bureau of Ships *ad interim* Specifications 46S18 (5).
[b] Bureau of Ships *ad interim* Specifications 14-0-15 (6).
[c] McClendon, Gault, and Mulholland (7).
[d] Subow (8).
[e] Lyman and Fleming (9).

made evident in Table III where ion concentrations are listed for each solution and in Table IV which presents the Cl-ratios. Only the proposed formula provides a solution having Cl-ratios which are identical with those of an average ocean water. Of the others, that of Lyman and Fleming (9) most nearly approaches the composition of natural ocean water. The formulas, 46S18 and 14–0–15, are noticeably different, especially because of the absence of bicarbonate ion which is most important in determining the pH of ocean water and in galvanic corrosion in ocean water.

TABLE III.—ION CONCENTRATIONS IN NATURAL AND SYNTHETIC OCEAN WATERS.

Ion	Grams per Liter at 20C.						
	46S18	14–0–15	M–G–M	Subow	L and F	Proposed	Natural
Na^+	10.20	11.150	11.336	10.765	10.83	11.0347	11.0347
Mg^{++}	1.317	1.33	1.263	1.325	1.303	1.3297	1.3297
Ca^{++}	0.403	0.447	0.431	0.422	0.408	0.4184	0.4184
K^+	0.400	0	0.5376	0.389	0.3833	0.3972	0.3972
Sr^{++}	0	0	0	0	0.0136	0.0139	0.0139
Cl^-	17.952	19.76	18.414	19.445	19.440	19.8405	19.8405
SO_4^{--}	2.400	2.74	2.653	2.708	2.751	2.7687	2.7687
Br^-	0.604	0	0.0461	0.057	0.0661	0.0675	0.0675
I^-	0	0	0	0	0	0	0.00005
F^-	0	0	0	0	0.0014	0.00136	0.00136
HCO_3^-	0	0	0.1475	0.1501	0.1428	0.1460	0.1460
H_3BO_3	0	0	0.0594	0	0.0266	0.0272	0.0272
Chlorinity	17.55	19.27	19.0	19.0	19.0	19.381	19.381

TABLE IV.—Cl-RATIOS IN NATURAL AND SYNTHETIC OCEAN WATERS.

Ion	46S18	14–0–15	M–G–M	Subow	L and F	Proposed	Natural
Na^+	0.5675	0.5650	0.583	0.5535	0.557	0.5556	0.5556
Mg^{++}	0.0733	0.0674	0.0649	0.0681	0.0670	0.06695	0.06695
Ca^{++}	0.0224	0.0227	0.0222	0.0217	0.02097	0.02106	0.02105
K^+	0.0203	0	0.0276	0.0200	0.0197	0.0200	0.0200
Sr^{++}	0	0	0	0	0.0007	0.0007	0.0007
Cl^-	0.9989	0.9987	0.9470	0.9995	0.9990	0.99895	0.99895
SO_4^{--}	0.1337	0.1388	0.1364	0.1392	0.1413	0.1394	0.1394
HCO_3^-	0	0	0.00758	0.0078	0.00683	0.00735	0.00735
Br^-	0.0336	0	0.00237	0.0029	0.00340	0.00340	0.00340
I^-	0	0	0	0	0	0	0
F^-	0	0	0	0	0.000072	0.00007	0.00007
H_3BO_3	0	0	0.00306	0	0.00137	0.00137	0.00137

Experience has shown that when the proposed solution is being used in large quantities over a long period of time, it can best be prepared with the components, other than sodium chloride and sodium sulfate, being added from three stock solutions. These stock solutions are given in Table V. To prepare a sample of the solution, 200 ml. of stock solution No. 1 and 100 ml. of stock solution No. 2 are added to several liters of distilled water in which have been dissolved 245.34 g. of sodium chloride and 40.94 g. of anhydrous

TABLE V.—STOCK SOLUTIONS FOR PREPARING SYNTHETIC SEA WATER.

(grams per liter).

No. 1	
$MgCl_2 \cdot 6H_2O$	555.6
$CaCl_2$ (anhydrous)	57.93
$SrCl_2 \cdot 6H_2O$	2.11
No. 2	
KCl	69.45
$NaHCO_3$	20.10
KBr	10.05
H_3BO_3	2.72
NaF	0.30
No. 3	
$Ba(NO_3)_2$	0.0994
$Mn(NO_3) \cdot 6H_2O$	0.0546
$Cu(NO_3)_2 \cdot 3H_2O$	0.0396
$Zn(NO_3)_2$	0.0151
$Pb(NO_3)_2$	0.0066
$Ag(NO_3)_2$	0.00049

sodium sulfate. This is diluted to a final volume of 10 l. and the pH is adjusted to 8.2 by the addition with stirring of 0.1 *N* sodium hydroxide solution. When heavy metals are desired, 10 ml. of stock solution No. 3 are added with stirring to the 10 l. of solution.

Stock solution No. 3 is prepared with nitrate salts of the heavy metals. Its addition or omission therefore will not affect the Cl-ratios of the more abundant ions. The nitrate ion introduced by this stock solution will be present within the normal limits of nitrate ion in natural ocean water.

SPECIFICATIONS FOR SALT SPRAY CORROSION TESTS

A.S.T.M. Tentative Method B 117 – 44 T[4] specifies a continuous spray of 20 per cent sodium chloride solution at 92 to 97 F. at a certain spray density. This spray density, defined in terms of volume of liquid settling from the spray atmosphere into a 10-cm. funnel, is restricted to 0.5 to 3.0 ml. per hour. The specification does not establish limits for the duration of tests under these conditions nor does it offer a system for interpreting the results obtained. Modifications of this specification prescribe lower concentrations of sodium chloride or solutions called synthetic sea water such as those given under 46S18 and 14–0–15 in Table II.

Generally speaking, these specifications do not interpret test results directly in terms of service performance. This is proper, but occasionally such unjustified liberties have been taken with the result that the salt spray test method has been the target of considerable adverse criticism. However, since the corrosive agent and the test conditions usually employed in spray cabinets are so different from those encountered in natural environments, it was not considered likely that the corrosion which occurs in spray cabinets would closely parallel that experienced in natural environments. Should it develop that there is some parallel performance, it would be expected that tests with the proposed substitute for natural ocean water would be most likely to produce results like those observed in natural environments. In order to develop some data to clarify this situation a number of comparative tests have been conducted.

CORROSION TEST CONDITIONS

Comparative tests were conducted in four identical spray cabinets using natural water from the ocean at Kure Beach, N. C., the proposed solution, 3 per cent and 20 per cent sodium chloride. The spray cabinets were air-jacketed and operated according to A.S.T.M. Method B 117 – 44 T, namely, 92 to 97 F., spray density of 0.5 to 1.0 ml. per hour in a 10-cm. funnel type of collector, and in the case of 20 per cent sodium chloride at 18 to 22 per cent as indicated by the gravity of collected samples. The concentrations of 3 per cent sodium chloride spray and of the natural and synthetic ocean waters were controlled within 10 per cent of the nominal concentration.

Suspended solids in the natural ocean water would clog the spray nozzles within one or two hours after start of the spray cabinet. It was therefore necessary to pass the natural ocean water through a sand filter before introducing it into the spray cabinet. Furthermore, to insure a fresh supply of natural ocean water, it was allowed to flow through the reservoir of the spray cabinet at a low rate. Other solutions were handled in the manner usual for 20 per cent sodium chloride solutions.

Test specimens also were exposed in the natural atmosphere at Kure Beach in two locations, 80 ft. and 800 ft. from the ocean, and were immersed in the ocean at the same location.

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[4] Tentative Method of Salt Spray Testing, 1949 Book A.S.T.M. Standards, Part 2, p. 786.

Preliminary experiments with mild steel in the spray cabinets using 20 per cent sodium chloride and the synthetic sea water showed that care must be used in selecting the angle of repose of specimens. Steel specimens, 3 by 4 in., were painted on one side and then exposed for 24 hr. at several angles.

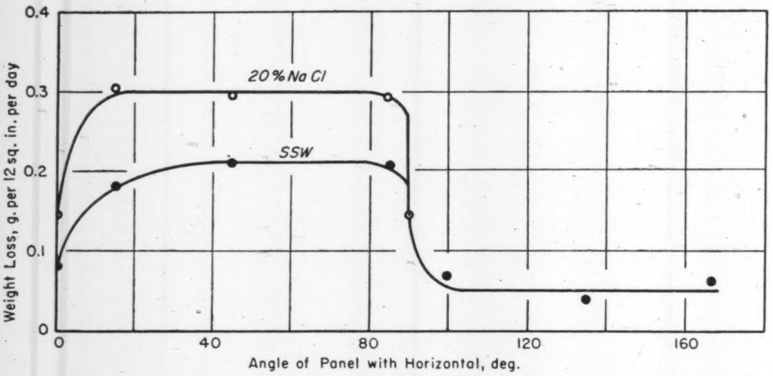

FIG. 1.—Effect of Specimen Angle on Corrosion in Salt Spray Cabinets Using 20 per cent Sodium Chloride and Synthetic Sea Water.

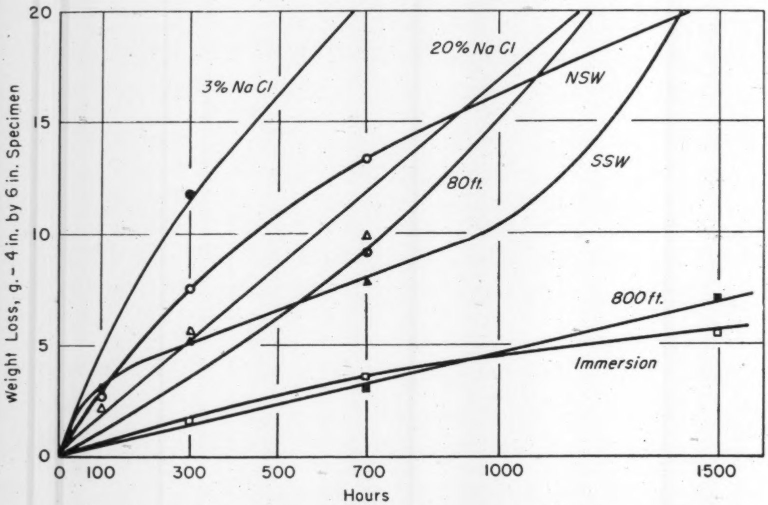

FIG. 2.—Comparative Tests on Ingot Iron (0.04 per cent Cu) in Sprays of Natural Sea Water (NSW), Synethetic Sea Water (SSW), 3 per cent Sodium Chloride, 20 per cent Sodium Chloride, in the Atmosphere 80 and 800 ft. from the Ocean and Immersed in the Ocean.

Figure 1 shows that nearly all corrosion occurs on the upper side of inclined test specimens and that the corrosion rate is nearly constant between 40 and 80 deg. with the horizontal. Accordingly, all specimens in the spray cabinets were exposed at an angle of 70 deg. with the horizontal. Atmospheric specimens were exposed at 30 deg. with the horizontal and sea water immersion specimens were vertical.

Sea water immersion specimens were 4 by 12 in. with two ¾-in. holes on 10-in. centers for mounting. The atmospheric and spray cabinet specimens were 4 by 6 in. All specimens were of either No. 16 or 20 gage thickness. Each test was conducted in quadruplicate.

The spray cabinet tests of bare metals ran for 100, 300, 700, and 1500 hr., atmospheric specimens for 700 and 1500 hr., 6 and 18 months. Sea water immersion tests ran 300, 700, 1500 hr. and 6 months. Specimens with metallic coatings were exposed until breakdown of the coating occurred.

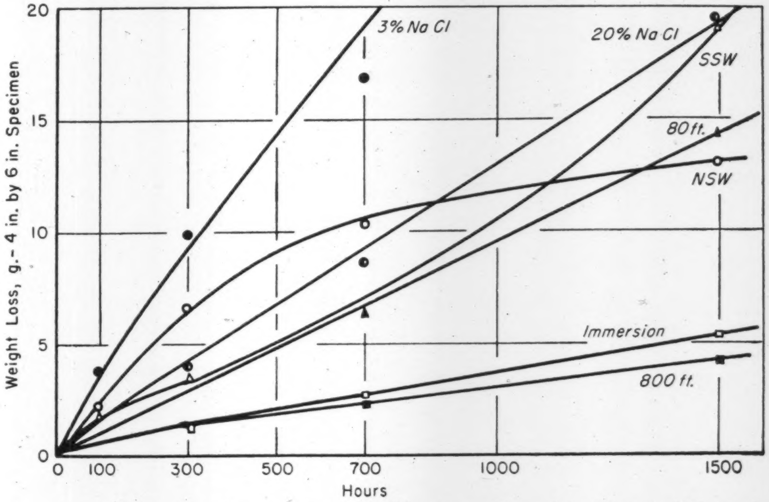

FIG. 3.—Comparative Tests on Steel (0.05 per cent Copper) in Sprays of Natural and Synthetic Sea Water, of 3 per cent and 20 per cent Sodium Chloride, in the Atmosphere 80 and 800 ft. from the Ocean, and Immersed in the Ocean.

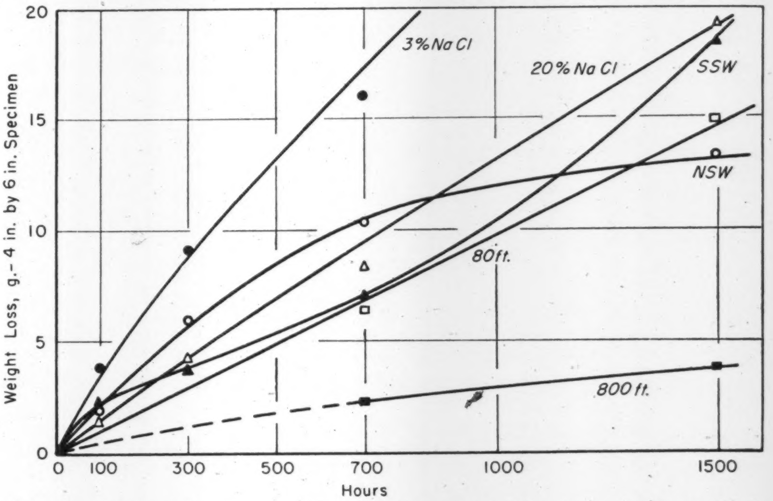

FIG. 4.—Comparative Tests on Steel (0.27 per cent Cu) in Sprays of Natural and Synthetic Sea Water, of 3 per cent and 20 per cent Sodium Chloride, in the Atmosphere 80 and 800 ft. from the Ocean, and Immersed in the Ocean.

Materials on which tests have been

completed and the results herein reported include the following:

1. Ingot iron, 0.04 per cent copper.
2. 0.05 per cent copper steel.
3. 0.27 per cent copper steel.
4. Rolled zinc, 0.065 per cent lead, 0.009 per cent iron, 0.0035 per cent cadmium.
5. 0.001 in. electrodeposited zinc on 0.14 per cent copper steel.
6. 0.001 in. electrodeposited nickel on 0.14 per cent copper steel.
7. Titanium.

Whenever possible corroded specimens were cleaned by scrubbing without an abrasive. The corrosion product on zinc specimens was loosened by a short immersion in ammoniacal ammonium chloride solution and then scrubbed off. Cathodic cleaning in inhibited sulfuric acid was employed on steels wherever the corrosion product could not be removed by scrubbing alone.

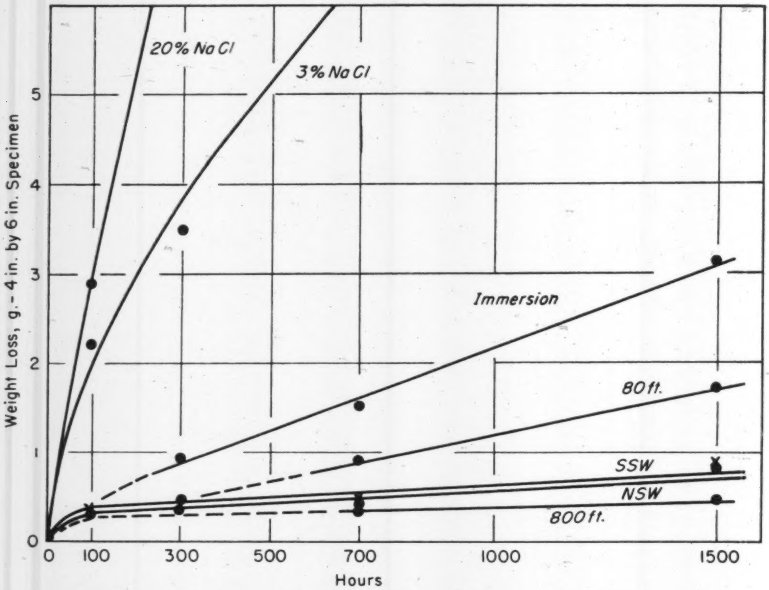

FIG. 5.—Comparative Tests on Zinc in Sprays of Natural and Synthetic Sea Water, 3 per cent and 20 per cent Sodium Chloride, in the Atmosphere 80 and 800 ft. from the Ocean, and Immersed in the Ocean.

RESULTS OF CORROSION TESTS AND DISCUSSION

Figure 1 shows the weight loss which occurred only on one side of each test panel since one side was protected by paint. In Figs. 2 through 5, the weight losses represent the total from both sides of each test specimen.

Titanium:

Tianium suffered no apparent corrosion during 6000 hr. in the spray cabinets and during longer periods in the natural environments at Kure Beach.

Iron and Steel:

In the spray cabinets, corrosion was confined almost entirely to the upper surface of the test specimens until after 300 hr. In exposures beyond this time corrosion on the lower side set in at the upper edge and then progressively spread over the surface. In contrast to this, specimens in the atmosphere suffered corrosion on both sides from the very start, the lower side seeming to suffer the more severe attack. Specimens immersed in sea water were uniformly corroded on both sides.

There was a very marked difference in the nature of the corrosion products on iron and steel in the different test conditions. Spray tests with natural and synthetic sea water produced grayish-white deposits on the upper surface which were mottled with patches of iron rust. The specimens exposed to 3 per cent and 20 per cent sodium chloride spray rapidly acquired a coating of iron rust which appeared to be hydrous ferric oxides. Between this and the metal was a layer of black ferrous oxides. These corrosion products were quite similar to those formed on iron and steel immersed in sea water. The only difference seemed to be in a tendency of the ferric corrosion products to form a loosely adherent scale or crust in sea water, whereas no crust formation occurs in the spray cabinets. In the natural atmosphere, the iron and steel corroded to form the usual laminated corrosion products which are less hydrated than in the spray cabinets or in sea water.

Figure 2 serves to compare the corrosion of ingot iron in the various environments. It is noted that 3 per cent sodium chloride spray is the most corrosive, whereas the atmosphere 800 ft. from the ocean and immersion in sea water were least corrosive. Confining attention to results below 700 hr., the other conditions do not present a clear pattern. The tests in synthetic sea water exhibited a sharp decrease in corrosion after 100 hr. which was not sustained. Spray with natural sea water indicated a slight protective effect from the deposits formed on the surface. The 20 per cent sodium chloride spray, like the 3 per cent sodium chloride spray, produces a very slight decrease in corrosion rate with time. These data on ingot iron indicate the corrosive order of these environments to be (1) 3 per cent sodium chloride spray, (2) natural sea water spray, (3) 20 per cent sodium chloride spray, (4) synthetic sea water spray, (5) atmosphere 80 ft. from the ocean, (6) atmosphere 800 ft. from the ocean, and (7) immersion in sea water.

Tests with two steels, containing 0.05 per cent copper and 0.27 per cent copper, produced very similar results as illustrated in Figs. 3 and 4. The order of corrosivity is the same for each of the steels. Only slight differences were noted in their corrosion rates, ingot iron usually exhibiting the greater weight losses.

Zinc:

In contrast to the situation with steel, 20 per cent sodium chloride spray is more corrosive than 3 per cent. These spray solutions in turn are very much more corrosive than any of the other environments in these tests. The sprays with natural and synthetic sea waters produce essentially the same corrosion rates and are slightly more corrosive than the atmosphere 800 ft. from the ocean. The atmosphere 80 ft. from the ocean and sea water immersion cause more rapid attack than the exposures in sprays of natural and synthetic sea water. Figure 5 presents these data.

The high rates of attack in the sprays of sodium chloride may be explained by the nature of the corrosion products. A non-protective, gelatinous zinc hydroxide is formed in the 3 per cent and 20 per cent sodium chloride sprays. In sprays of synthetic and natural sea water, the corrosion product is a hard, granular and adherent coating which is quite protective. It is similar to the corrosion product formed on zinc immersed in sea water and probably is of similar composition, namely, a mixture of basic zinc carbonate, sulfate and chloride. The corrosion product on zinc in the atmosphere 800 ft. from the ocean was softer than that in the latter spray cabinets and had a chalky texture. However it was somewhat protective. In the atmosphere 80 ft. from the ocean the corrosion products were less chalky and tended to be granular although not as granular as formed in the sprays of natural and synthetic sea water.

If one notes that the corrosion occurs only on one side of specimens in the spray cabinet, it becomes apparent that the corrosion rate per unit of exposed surface is about the same order of magnitude for the spray test with natural and synthetic waters and the atmosphere 80 ft. from the ocean. The spray tests with sodium chloride solutions produce an entirely different intensity and type of corrosion which does not duplicate normal marine corrosion of zinc.

Zinc-Plated Steel:

Zinc that has been electrodeposited on steel, initially behaves like the rolled zinc already described. As the coating is corroded away rust stains appear. In sea water, the four specimens did not provide good checks, failing in 1, 3, 4, and 6 months, respectively. In the atmosphere, only slight rust staining appeared at the edges in contact with the porcelain insulators in 9 months. This staining progressed only slightly during the following 5 months. These one-mil coatings of zinc failed in 50 days in 20 per cent sodium chloride spray and in 90 days in 3 per cent spray. In synthetic and natural sea water sprays, slight rust staining developed on the edges in 90 days. This showed very slight change during the next 90 days at which time the exposure was terminated.

It would appear that the natural and synthetic sea water sprays provide a close parallel to the natural atmosphere exposures. The sodium chloride sprays do not compare with the natural environments in the type of corrosion encountered.

Nickel-Plated Steel:

These specimens with 0.001 in. of gray nickel on steel produced very interesting results. In sea water, all specimens developed rust stains in 24 hr., and the 0.06-in. steel specimens perforated in 14 days at some of these pinholes. The spray cabinets produced a similar result. Failures at a few large pinholes became evident in 24 hr. in all cabinets. All specimens failed in 7 days, and it is difficult to say which was the most corrosive. A general observation would place the spray solutions in order of decreasing corrosivity as (1) 20 per cent sodium chloride, (2) 3 per cent sodium chloride, and (3) natural and synthetic sea water. In the atmosphere, numerous small pinholes appeared with light rust staining around them. In 165 days, there was little difference between specimens located 80 and 800 ft. from the ocean. At this time they were given an approximate rating of 2 to 3 according to the system for rating chromium-plated steel used by Committee B-8.[5]

The great difference in attack on nickel-plated steel in the cabinets and in the atmosphere may be readily explained. In the cabinets and in sea water, where the surfaces are continuously wet, the first pinholes to appear in the coating expose steel which then serves as an anode to protect cathodically the remaining intact nickel surface. Hence only a few pinholes develop and these exhibit a severe attack. In the atmosphere, the specimens are not continuously wet by a conducting solution and the coating does not receive cathodic protection from the early breaks in the coating. Therefore, a large number of pinholes develop in each of which the corrosion is slight in comparison to the specimens continuously wet with conducting solution.

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[5] Report of Subcommittee II on Performance Tests, A.S.T.M. Committee B-8 on Electrodeposited Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 220 (1949).

Because of this radical difference, it is questionable as to what can be said about the behavior of nickel-plated steel in the atmosphere on the basis of salt spray tests. On the other hand, sea water immersion and spray cabinet tests produce the same type of failure, with the latter being the less severe exposure.

SUMMARY

A formula has been proposed for a solution which duplicates the inorganic components of natural ocean waters. Comparative tests have been conducted in spray cabinets using 3 per cent and 20 per cent sodium chloride, natural ocean water, and the proposed solution. At the same time corrosion tests have been conducted in the natural environments at Kure Beach, N. C.

These tests reveal little similarity of performance between the natural environments and the spray cabinets using 3 per cent and 20 per cent sodium chloride solution. The only exception exists in the deterioration of nickel-plated steel immersed in sea water in which case all spray tests caused a similar type of failure.

In tests with ingot iron and plain steels, the spray tests with natural and synthetic sea waters did not duplicate the types of corrosion usually observed in natural marine environments.

Zinc-plated steel and solid zinc sheet corrode in a similar manner in spray cabinets using natural and synthetic sea waters and in the natural environments at Kure Beach. However, the corrosion rates were not sufficiently high to warrant considering such cabinet tests as accelerated tests.

Acknowledgment:

The authors wish to acknowledge the assistance of H. T. Paterson, Jr., W. C. Sierichs, and C. E. Black in the collection of some of the data and information reported in this paper.

The authors thank those persons who kindly furnished materials for these tests, namely, O. B. Ellis, Armco Steel Corp., ingot iron; C. P. Larrabee, Carnegie Illinois Steel Co., 0.05 per cent copper and 0.27 per cent copper steel; E. A. Anderson, New Jersey Zinc Co., rolled zinc; W. Blum, National Bureau of Standards, electroplated steel specimens; and G. E. Hutchinson, Remington Arms Co., Inc., titanium.

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DISCUSSION

Mr. Alfred H. Woodcock.[1]—My experience in sampling atmospheric sea salt particles leads me to suppose that corrosion, resulting from the presence of these particles on steels exposed to the free air, would occur at a great many small points. This supposition is based entirely upon the fact that concentrated sea water aerosols are always deposited as separate droplets or crystalline masses. Is it possible that on the authors' plates each small point of corrosion marks the location of a drop which initiated this corrosion? I should like to ask also if the plates, which have been exposed to the free air, are dry in the cabinets? It was stated that the plates are not obviously wet. The presence of water would depend upon the relative humidity, since the sea-water drops on the plates are hydroscopic and will remain liquid at rather low relative humidities. Owens[2] found saturation occurring in drops of concentrated sea water at about 70 per cent relative humidity. Recently Dessens[3] indicated that atmospheric salt nuclei often remain liquid for many hours at humidities below 70 per cent.

Mr. F. L. LaQue.[4]—I merely wish to get into the record a suggestion that those concerned with the use of synthetic sea water in studying the behavior of materials in sea water under high-velocity conditions might refer to a paper by the present author and W. C. Stewart which appeared in the June, 1948 issue of *Metaux and Corrosion*, Vol. XXIII, No. 274, p. 147. Included in the studies covered by this paper were some erosion tests on bronzes in contact with a synthetic sea water made up in accordance with Dr. May's formula and in natural sea water at Kure Beach, N. C. By way of example, it was discovered that a bronze containing approximately 5 per cent each of tin, zinc, and lead corroded at a rate of only 87 mg. per sq. dm. per day in the synthetic sea water, as compared with a rate of 448 mg. per sq. dm. per day in the natural sea water. It was evident, therefore, that there are some constituents of sea water that have a profound effect on attack of materials at high-velocity which apparently are not readily incorporated in synthetic sea water. I suspect that these missing constituents are bacteria and other organisms capable of affecting corrosion and the nature of corrosion product films.

Mr. T. P. May (*author*).—In answer to Mr. Woodcock's first question, I agree that sea salt particles which may be deposited upon atmospheric test specimens are hygroscopic and therefore it is quite conceivable that in moderately dry weather these particles become the sites of small droplets of water. At other times when the humidity may be much higher or when an excessive amount of sea water spray is blown onto the specimens, the specimens may be covered with a continuous but thin film of sea water. In the first instance we would naturally expect corrosion to be confined to a large number of very small areas and would lead to the development of pinholes in the coating. In the second instance we would still expect the same result because the very thin film of liquid would introduce large electrolytic resistance to the flow of current from local anodes. Therefore, the development of rusting at a few pinholes will not be able to provide cathodic protection to the remaining surface. As a result the remaining surface will be susceptible to penetration and breakdown by the corrosive liquid on the surface. In contrast to these conditions which prevail in the atmosphere, specimens in the spray cabinets and those immersed in sea water are in contact with relatively thick films of water which have much lower electrical resistance.

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[1] Oceanographer, Woods Hole Oceanographic Institution, Woods Hole, Mass.

[2] J. S. Owens, "Condensation of Water from the Air upon Hygroscopic Crystals," *Proceedings*, Royal Soc. London, Series A, Vol. 110, pp. 738–752 (1926).

[3] Henri Dessens, "Brume et Noyaux de Condensation," *Annales de Geophysique*, Vol. 3, pp. 68–86 (1947).

[4] In Charge, Corrosion Engineering Section, The International Nickel Co., Inc., New York, N. Y.

They would provide protective currents to areas adjacent to the local areas of breakdown. These protective currents would prevent the development of new pinholes in the areas around the pinholes which form at the start of the exposure.

This discussion also answers Mr. Woodcock's second question concerning the point as to whether the specimens are dry in the test cabinets.

MR. T. J. SNODGRASS.[5]—I was curious that there seemed to be quite a good correlation on the zinc coatings in the salt spray and its other tests, and there was quite a close correlation between the 20 per cent sodium chloride salt fog and the immersion test.

Was that regular sea water immersion?

MR. MAY.—All immersion tests reported here were complete immersion of specimens in natural sea water. I am not convinced that there was good agreement between the tests in 20 per cent sodium chloride and in the sea water immersion. Severe corrosion occurred in the 20 per cent salt spray in 50 days, whereas failure of zinc-coated specimens in sea water showed a similar degree of failure in periods of 1 to 6 months. From this it is very indefinite as to what one would consider to be a failure for sea water immersion. If anyone can find a correlation between any of these exposures, I am quite confident that it cannot be accounted for in any logical manner. In fact any agreement that one does find in these tests must be considered purely accidental.

MR. SNODGRASS.—That correlation was not secured with nickel, nor with the bare steel specimens. In other words, the immersion was way down on the bare steel. Yet, with the zinc, of course, it was quite close to the 20 per cent. I am not thinking so much about the sea water, but there are many other applications where zinc-plated parts are used under immersion or alternate immersion conditions, and if the correlation between the 20 per cent salt fog and the immersion is that close, one might really have something.

MR. MAY.—In a particular case it may develop that corrosion as expressed by weight losses are more or less of the same order in the spray cabinets as in some other environment. However, if these environments differ in any respect, I must emphasize again that any good correlation will be accidental for any particular time interval. The correlation will not necessarily hold for extrapolation to other times and to other environments. Accordingly, one cannot depend on results in a spray test to tell him how long a zinc-coated steel will last in some other environment.

MR. LAQUE.—It does not seem particularly advantagenous to use an accelerated test to get a rate of corrosion which could be measured directly. I would be inclined to take the direct approach and make the test under natural conditions if I were interested in immersion corrosion.

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[5] Appliance and Merchandise Dept., General Electric Co., Trenton, N. J.

SPRAY TESTING WITH NATURAL AND SYNTHETIC SEA WATER. PART II.—A STUDY OF ORGANIC COATINGS*

BY ALLEN L. ALEXANDER[1] AND THOMAS P. MAY[2]

As a logical extension of the work reported in the previous paper[3] experiments were conducted in which a variety of organic coatings applied to several metals was exposed simultaneously to the spray of natural and synthetic sea water and to 3 per cent and 20 per cent sodium chloride solutions. These experiments were decided upon following a joint meeting of the Test Methods and Corrosion Sub-Committees of the JAN Prevention of Deterioration Committee in September, 1946, at which time the formula for the synthetic sea water was first presented to a sizable group interested in the standardization of test procedures involving simulated sea water. Shortly following the initial organization of these experiments, the original committee was dissolved, but the tests have continued through a cooperative arrangement from which the present material has become available.

The salt spray test has been used over a long period in a variety of modifications in attempts to assess the durability or resistance to degradation of numerous materials required to withstand corrosive attack by natural forces prevailing in or adjacent to the sea. The use of brine solutions varying between 3 and 20 per cent in salt-fog cabinets is rather common. May[4] has shown that marked differences exist in the rate of corrosion of mild carbon steel—as well as in the type of corrosion product—when exposed to synthetic sea water and salt fog of varying concentration. Inasmuch as the results of salt spray and exposure to synthetic sea water and fog are used frequently to indicate acceptability for use in marine environment, it appeared of interest to study the behavior of a number of items normally subjected to the salt spray test when exposed simultaneously to sea water, sea water fog, synthetic sea water fog, to brine fog of at least two concentrations and to marine atmospheres. The results obtained with a variety of metals and plated coatings are the subject of another paper.[3] The data presented here and discussed in some detail are confined to a study of organic coatings applied to metal.

EXPERIMENTAL

Reference is made to the preceding paper[3] relative to the details of experimental conditions existing within the boxes for the duration of the experiments. The boxes were of identical standard design known as type CA-2, produced by the Industrial Filter and Pump Co. They were placed in position such that one box could take its supply of sea water by passing through a

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Protective Coatings Branch, Chemistry Division, Naval Research Laboratory, Washington, D. C.

[2] Corrosion Engineering Section, Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

[3] T. P. May and A. L. Alexander, "Spray Testing with Natural and Synthetic Sea Water, Part I.—Corrosion Characteristics in the Testing of Metals," see p. 1131 this publication.

[4] T. P. May and C. E. Black, "Synthetic Ocean Water," *NRL Report No. P-2909*.

TABLE I.—GROUP A.

System	Sea Water	Synthetic Sea Water	3 per cent Brine	20 per cent Brine	Immersion	80-ft. Rack	800-ft. Rack
FIRST INSPECTION—42 DAYS							
A	10	10	10	10	10	10	10
B	7	7	6	8	7	10	10
C	9	9	8	10	8	10	10
D	9	9	8	9	9	10	10
E	9	9	8	9	9	10	10
SECOND INSPECTION—92 DAYS							
A	10	10	10	10	9	10	10
B	5	5	4	5	2	9	10
C	6	7	1	8	7	10	10
D	8	9	5	6	9	10	10
E	9	8	5	6	9	10	10
THIRD INSPECTION—152 DAYS							
A	10	10	10	10	7	10	10
B	5	5	3	2	1	8	10
C	2	4	0	4	7	10	10
D	5	5	2	4	8	9	10
E	8	8	3	4	9	9	10

minimum of pipe. Fog density, temperature, and position of the panels were checked periodically and maintained as nearly constant as practical. The panels were supported from the bottom in plastic racks at an angle of 70 deg. to the horizontal, at positions sufficiently far apart that the condensate on one would not fall onto another.

Since this experiment was designed as a study of a method rather than an investigation of the relative properties of a number of coatings, a cross-section representing a variety of formulations was selected for inclusion. Systems normally applied to aluminum and magnesium surfaces of aircraft were studied, along with selected materials specified for steel in a number of varied applications. The systems are identified as follows:

System A:

Material	24S-T aluminum alloy anodized
First coat	Zinc chromate primer (AN-TT-P-656b)
Second coat	Zinc chromate primer (AN-TT-P-656b)
Third coat	Gray Camouflage lacquer (AN-L-21)
Fourth coat	Gray camouflage lacquer (AN-L-21)

System B:

Material	Magnesium AN-M-12 Type II chrome pickle
First coat	Zinc chromate primer (AN-TT-P-656b)
Second coat	Zinc chromate primer (AN-TT-P-656b)
Third coat	Gray camouflage lacquer (AN-L-21)
Fourth coat	Gray camouflage lacquer (AN-L-21)

For systems C, D and E, cold-rolled steel formed the base metal which was given a treatment with Inhibitive Wash USA Spec. 3-213 prior to painting.

System C:

	gal.	lb.
First and Second Coats		
Red iron oxide	7.75	327.8
Zinc chromate	5.05	147.8
Diatomaceous silica	1.48	32.7
Magnesium silicate	3.46	82.2
Vehicle temperature V-1	82.26	616.5
	100.00	1207.0
Third and Fourth Coats		
Red iron oxide	9.21	348.0
Silica	1.56	34.5
Magnesium silicate	3.63	86.1
Vehicle temperature V-1	85.60	646.4
	100.00	1115.0

System D:

	gal.	lb.
First and Second Coats		
Aluminum paste	13.5	165
Zinc chromate	4.2	123
Vehicle temperature V-1	82.3	622
	100.0	910
Third and Fourth Coats		
Aluminum paste	14.1	172
Vehicle temperature V-2	85.9	660
	100.0	832

System E:

	gal.	lb.
First and Second Coats		
Aluminum paste	13.5	165
Zinc chromate	4.2	123
Vehicle temperature V-1	82.3	622
	100.0	910
Third and Fourth Coats		
Aluminum paste	14.1	172.0
Vehicle temperature V-1	85.9	649.5
	100.0	821.5

NOTE.—Vehicle V-1—Dark phenolic resin tung oil varnish.

Vehicle V-2—Phenolic tung-linseed varnish.

In systems F through J, cold-rolled steel formed the base metal which was solvent-cleaned before priming.

System F:
1 coat WP-1 primer
1 coat P-21 vinyl primer
2 coats vinyl alkyd topcoat

System G:
1 coat 52-P-18 primer
1 coat 52-P-45 haze gray topcoat

System H:
1 coat WP-1 primer
1 coat 52-P-18 primer
1 coat 52-P-45 haze gray topcoat

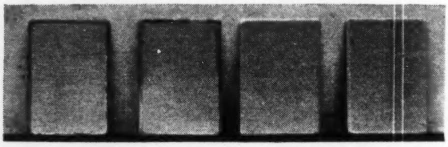
System I:
1 coat WP-1 primer
1 coat 656b primer
2 coats AN-E-3 gray enamel

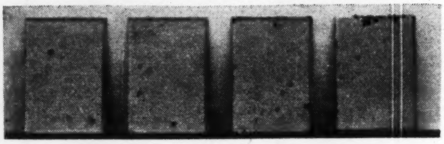
System J:
1 coat 656b primer
2 coats AN-E-3 gray enamel

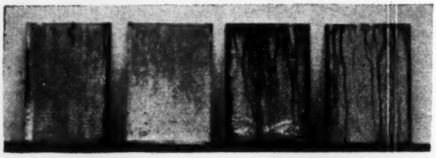
NOTE.—All topcoats approximate 2.2 mils in thickness.

Systems A, B, and F through J were prepared by the Naval Research Laboratory; systems C, D, and E were finished by the Engineer Research and Development Laboratory at Ft. Belvoir, Virginia, from the steel supplied from the same lot at NRL. The steel for the painted panels, the panels electroplated at the Bureau of Standards, and the bare steel panels (described previously) were all from the same lot. The painted specimens were inspected regularly at one- to three-month intervals by personnel from Ft. Belvoir and NRL, at which time all panels were photographed. The systems selected represented typical applications for aircraft and ships, along with a few experimental formulations of a type that might be considered for use on motorized field equipment.

The results of the first inspection are shown in Table I, where a rating of 10 signifies the film to be unaffected and 0 implies complete failure. System A, the standard aircraft finish on aluminum alloy, is unaffected after 42 days, whereas the identical system applied over magnesium (system B) has permitted considerable failure. The result of immersion appears to be similar in effect to the spray, while nothing detectable has occurred from exposure to the atmosphere at either 80 ft. or 800 ft. from shore. Regarding severity of attack, the similarity between sea water and synthetic sea water is striking; and 3 per cent brine is the most severe, while the 20 per cent solution is somewhat less damaging. The data of Table I describe the condition of the films at the end of 92 days in which the same general pat-


System A.


System B.


System C.

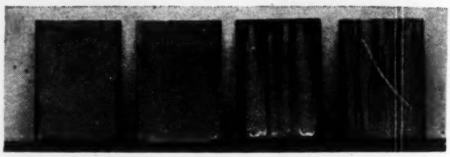
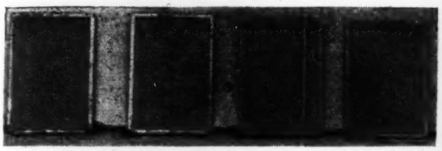
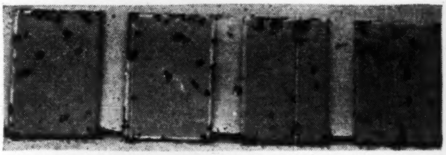

System D.

FIG. 1.—92 Days Exposure.



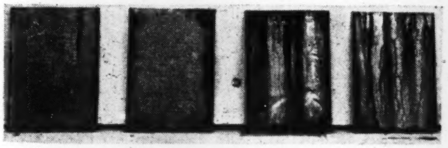
System A.

System B.

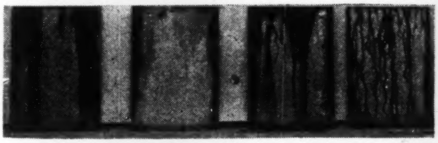


Replicates Box No. 2.

System C.



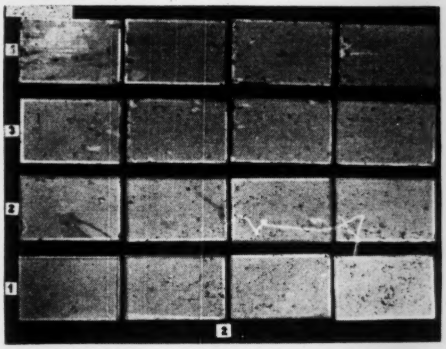
System D.



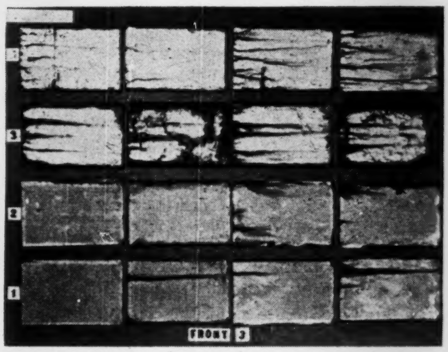
System E.

FIG. 2.—152 Days Exposure.

tern is maintained, with sea water, perhaps, being perceptibly more destructive than the synthetic variety. As before, the corrosivity of the brine solution becomes increasingly evident—particularly for the coatings over steel. One panel from each exposure and system



System B.

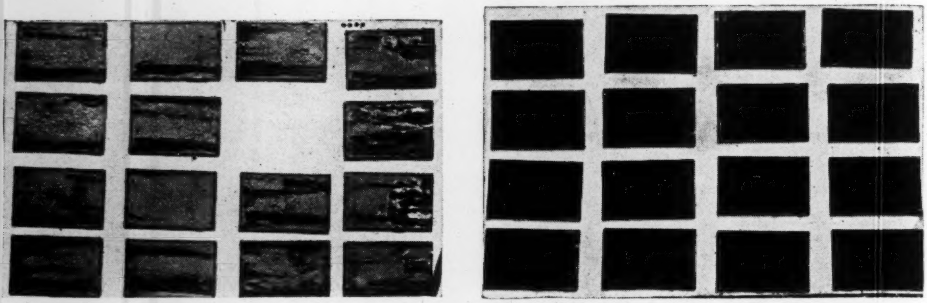


System C.

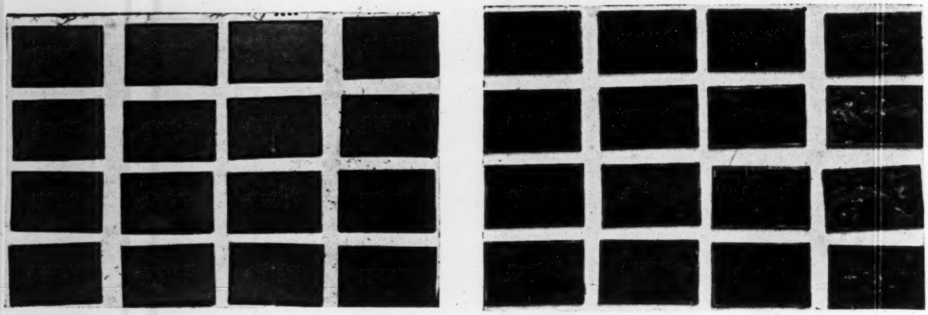
FIG. 3.—Replicate Samples.

TABLE II.—GROUP B.

System	Sea Water	Synthetic Sea Water	3 per cent Brine	20 per cent Brine
SECOND INSPECTION—83 DAYS				
F	10	10	10	10
G	1	4	2	3
H	9	9+	7	9+
I	5	6	4	5
J	1	2	0	0



System G. System I.



System F. System J.

FIG. 4.—Comparison of Systems F, G, I, and J.

is shown in Fig. 1 in the order (left to right), sea water, synthetic sea water, 3 per cent brine, and 20 per cent brine. After 152 days, the bulk of group A (first series) was terminated and the final evaluation made, as shown in Table I. Again, the striking similarity between natural and synthetic sea water is noted and, once more, the marked severity of the brine solutions is apparent. The results of total immersion in the sea and atmospheric exposures are included for comparative purposes.

From this final inspection a number of conditions exist that deserve some comment. The standard aircraft system (two coats primer followed by two coats lacquer) is sufficient to protect aluminum for considerable periods when exposed to any of the sprays studied. Equal protection is not afforded to continuously submerged specimens. Therefore, an immersion test is likely to be more conclusive than a spray test for evaluating coatings applied to aluminum where time is a factor. As was expected, the same organic system fell far short of protecting magnesium in the same circumstances. Brine solutions are more corrosive to painted magnesium than sea water of either variety. One panel from each box, in the usual order, is shown in Fig. 2. The close agreement between replicate samples may be seen in Fig. 2, which shows all four samples of painted magnesium upon removal from the synthetic sea water spray. This agreement is typical of that obtained generally between replicate painted samples throughout the experiment. Figure 2 shows one panel from each box for systems C, D, and E, which augment the data of Table I. For each of the paint systems on steel, the spray tests are more severe than total immersion; with brine spray appearing more corrosive than either synthetic or genuine sea water. Figure 3 shows all panels from all boxes for systems B and C, respectively. Again, the fair agreement between replicate samples exposed simultaneously is illustrated, while simultaneously the relative effect of the various sprays can be estimated.

Following the conclusion of the first phase of the study of organic coatings (Group A—Systems A to E), a second series was prepared and placed in the spray boxes (Group B—Systems F to J), consisting of systems F through J, representing systems considered for application to ships' topsides, along with the previously described aircraft systems applied to steel. These tests were inspected periodically, but only the final results appear sufficiently conclusive for inclusion here. The final summary for group B is presented in Table II. Even though it is evident that wide differences exist in the performance rating of the several finishes, the degree of degradation resulting from each spray retains the same relationship as before. The brines are most severe, with synthetic sea water following sea water very closely.

In addition to the observations on the effect of the test solutions, this series affords an excellent opportunity to study the role of WP-1 primer as a base coat for painted steel. The superiority of system F over system G, and of system I over system J, may be attributable, entirely, to the presence of this primer. In Fig. 4 replicate samples from each box are in the vertical columns, while the horizontal rows consist of test specimens from each of the four spray media.

SUMMARY

From the data obtained, it appears that the use of simulated sea water is justified as a test medium in lieu of genuine sea water for use in salt spray testing. For long-term experiments, sea water appeared slightly more detrimental than the synthetic product. However, in the evaluation of organic coatings applied to ferrous surfaces, 3 per cent brine is the most severe of the solutions investigated, while 20 per cent brine is slightly less degrading. Of particular interest is the close agreement between replicate samples throughout the study. No wide differences were found between like samples subjected to the same conditions. Similarly, the degree of difference obtained from the individual spray cabinets remain relative throughout, lending greater credence to the reliability of the results.

Acknowledgment:

The contribution of Roy W. Hill in preparing a number of the panels and participating in the periodic inspections is gratefully acknowledged.

DISCUSSION

Mr. F. L. LaQue.[1]—I gathered from Mr. Alexander's remarks that he thinks there is an advantage in using the synthetic sea water over natural ocean water—an advantage of convenience and perhaps reliability of getting the same thing from time to time, if you are located near sources of pollution.

Does he think there is any point in going to the trouble of making up this very complicated solution, as against making up a 3 per cent sodium chloride solution and using that in the box?

Mr. A. L. Alexander (*author*).—I think a very conclusive result of this experience described here is that, as far as organic coatings are concerned, the 3 per cent brine will yield results a lot more quickly than either the synthetic sea water or the real thing.

As far as accelerated testing is concerned, perhaps that is what we want. On the other hand, I don't know whether I favor this kind of test, exactly, as a specification requirement, because from one laboratory to another, from my experience and what I have seen, it has been rather hazardous to say that one test here, even though it allegedly meets some specifications, is exactly the same as that carried out elsewhere. We like to think of the salt spray test simply as a means of comparing perhaps a new product to a standard, or for comparative purposes only.

Mr. LaQue.—Just to keep the record straight, my question was not meant to advocate the use of spray tests for any purposes, but, rather, to ask in a simple way, is there any sense of making up May's fancy formula? It takes one of our men half a day to do that, but only a short time to make the sodium chloride solutions.

Mr. Alexander.—The answer, briefly, is no. However, I don't think we could have answered that before these experiments.

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[1] In Charge, Corrosion Engineering Section, The International Nickel Co., Inc., New York, N. Y.

IMPACT DEPOSITION OF ATMOSPHERIC SEA SALTS ON A TEST PLATE*

By Alfred H. Woodcock[1]

Rusting of structural steels at Block Island, R. I., and at Kure Beach, N. C., has been attributed in major part to the presence of chlorides on the test plates.[2] These chlorides were presumably atmospheric sea salts from marine air which were deposited on the plates in some unspecified manner. It is the purpose of the present paper to indicate the weight and the number of sea-salt nuclei which are deposited on surfaces by impact during the passage of marine winds in the Block Island region. It is suggested that impact deposition of sea salt on test plates in this region may largely account for the presence of chlorides on these plates. Thus wind speed and direction are suggested as primary factors in modifying the rate and the area of deposition of atmospheric sea salt on exposed surfaces.

No reference to the deposition of aerosols by impact has been found in studies and texts about the corrosion of steels in the atmosphere,[2, 3, 4, 5] though Vernon[6] has demonstrated the importance of aerosols by testing corrosion rates in filtered and in unfiltered air. Deposition of sea salt may occur in marine winds having a wide range of relative humidity. The impact deposition of atmospheric sea salt and other aerosols on test plates during winds may be of significance in explaining variable "edge corrosion"[2] and the excess corrosion of plates sheltered from rain.[4, 7]

Little description of methods is given in this paper. Details of the impact method used in sampling atmospheric sea salts have been published by Woodcock and Gifford.[8]

Wind-Deposition of Atmospheric Sea Salt on Test Plates

Figure 1 shows a sketch of a rectangular (10 by 15-cm.) test plate, *A*, mounted on a wind vane in such a manner that the plate surface is held normal to the air stream. A removable 4 by 50-mm. glass slide was inserted in a slot, *B*, in the middle of the plate, the outer glass surfaces being parallel to and in the plane of the edge and face of the metal plate.

This plate and slide were exposed to the wind for 48 min. at a relative humidity of 65 per cent and at a site free from the influence of local surf (see Table I for further information about the sampling location). During this time another smaller glass surface, *C*, was exposed to the wind for 31 sec. After exposure these two slides were taken to the laboratory, where counts and measurements of the impinged sea-salt nuclei were made with a special microscope under controlled temperature and humidity conditions.[9]

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Oceanographer, Woods Hole Oceanographic Institution, Woods Hole, Mass.

[2] H. R. Copson, "A Theory of the Mechanism of Rusting of Low Alloy Steels in the Atmosphere," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, pp. 554–590 (1945).

[3] R. J. McKay and Robert Worthington, "Corrosion Resistance of Metals and Alloys," Am. Chemical Soc. Monograph, Reinhold Publishing Corp., New York, N. Y., pp. 1–492 (1936).

[4] C. P. Larrabee, "The Effect of Specimen Position on Atmospheric Corrosion Testing of Steel," *Transactions*, Electrochemical Soc., Vol. 85, pp. 297–306 (1944).

[5] "Corrosion Handbook," John Wiley and Sons, Inc., New York, N. Y., pp. 1–1188 (1948).

[6] W. H. J. Vernon, "Second Experimental Report of the Atmospheric Corrosion Research Committee," *Transactions*, Faraday Soc, Vol. 23, pp. 113–204 (1927).

[7] N. B. Pilling and W. A. Wesley, "Atmospheric Durability of Steel Containing Nickel and Copper," *Proceedings*, Am. Soc. Testing Mats., Vol. 40, pp. 643–657 (1940).

[8] A. H. Woodcock and Mary M. Gifford, "Sampling Atmospheric Sea-Salt Nuclei Over the Ocean," *Journal Marine Research*, Vol. 8, pp. 177–197 (1949).

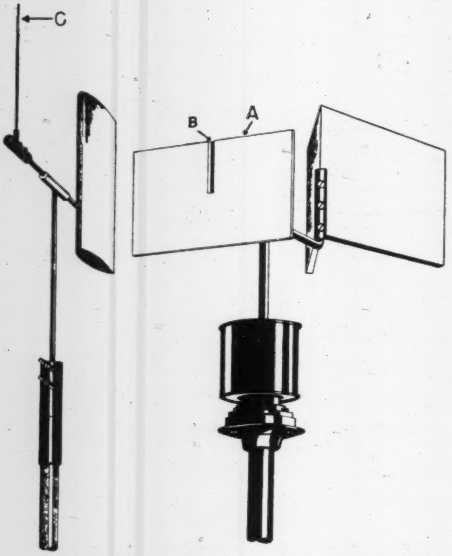

FIG. 1.—Diagrammatic Sketch of the Simple Devices Used to Expose Aerosol Sampling Surfaces to the Air Stream.

In Fig. 2 are shown photomicrographs of sea-salt nuclei on the two slides. These photographed areas of the slides were selected to show approximately the range of sizes present. The nuclei are shown as crystalline masses at a relative humidity of about 40 per cent, and as liquified and hemispheric drops at a humidity of 91 per cent. The weights of sea salt present in the hemispheric drops are computed from diameter measurements of these drops. This method of measuring atmospheric salt in impinged nuclei has been tested by titration of chlorides in nuclei similarly sampled on multiple surfaces.

On the 4 by 50-mm. slide on the test plate these nuclei deposited at an average rate, over the whole slide surface, of 6740 per hr. per sq. cm. (see Fig. 4 for the deposition rates of nuclei of the various weights). At this rate, deposition of sea salt in one day becomes about 3 mg. per sq. cm. (Table I). On the 1 by 15-mm. glass slide the nuclei impinged at a rate of 3.01 × 10^6 per hr. per sq. cm. The rate of deposition by weight on the smaller slide amounts to about 24 mg. of sea salt deposited per sq. cm. per day.

TABLE I.—ATMOSPHERIC SEA SALT SAMPLED AT THE SEAWARD END OF THE MARINE BIOLOGICAL LABORATORY PIER.

November 23, 1949. Time 1532 to 1620 EST. Average wind velocity 12.9 meters per sec., WSW. Sampling height 5 m. T_d 10.3 T_w 7.2 C. Nearest windward surf about 5 miles away.

	4 by 50-mm. Glass Slide on 10 by 15-cm. Plate					1 by 15 mm. Glass Slide
Distance from outer edge of plate, mm.	1	12.5	25	37.5	49	
Sea-salt deposition rate (mg. per sq. cm. per day.)	4.2	3.3	3.1	3.0	2.6	24.1
Number of nuclei measured	260	340	231	224	245	2705
Area on which counts were made, sq. mm.	4.58	6.75	4.40	4.36	4.51	4.70
Covering fraction	0.046	0.037	0.037	0.036	0.033	0.008
Exposure duration, sec.	2880					31
Exposure length, meters	36 529					400

Figure 3 shows the numbers of nuclei of the various weights which were counted and measured. The X's represent 1299 nuclei on the 10 by 15-cm. plate and the solid dots represent 2705 nuclei on the 1-mm. wide glass strip. Figure 4 shows the rates at which nuclei, of the various weights, impinged on the two slides. It is clear, as indicated in Fig. 4, that deposition is far more rapid upon the smaller (1 by 15-mm.) surface. Also it appears that this difference in rate becomes less with increasing weight of nuclei. Figure 5 shows the number of nuclei per cubic meter of air as indicated by the deposition on the two surfaces.

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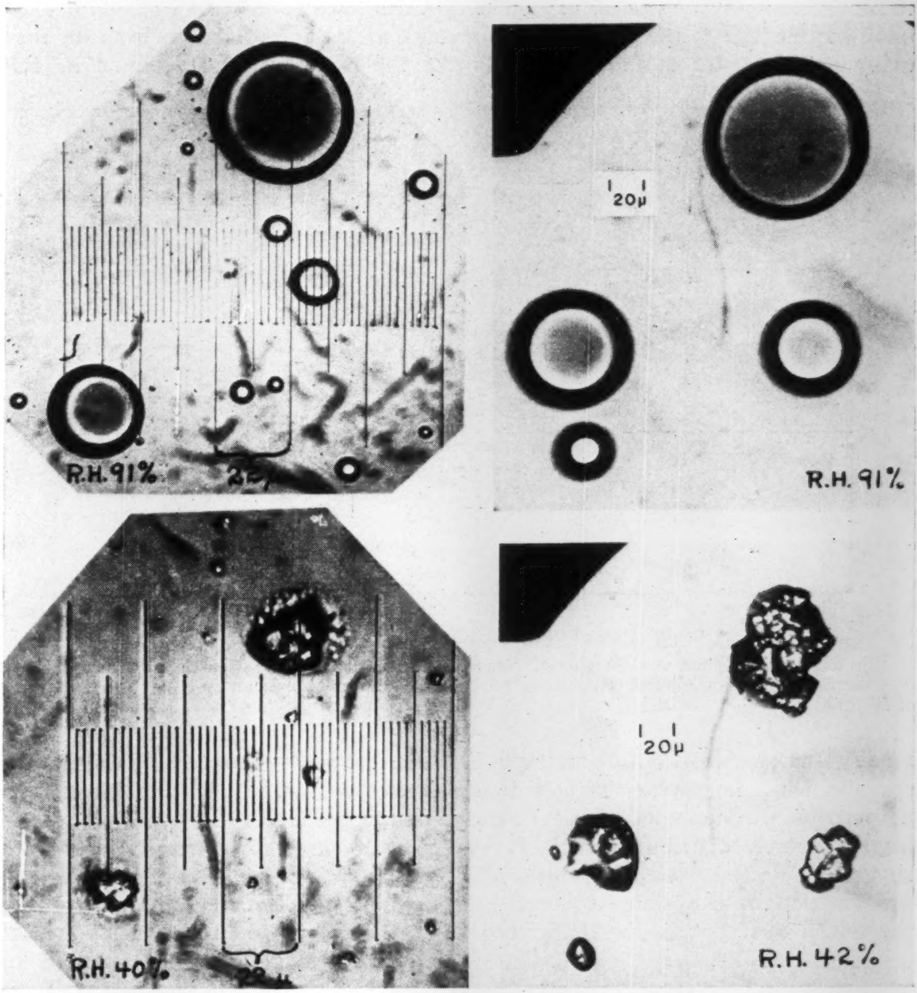
[9] The equipment used to obtain these measurements and the data presented in Figs. 7 and 8 became available to the author under Contract N6onr-277 with the Office of Naval Research.



Sea-Salt Nuclei Deposited on Glass Strip 1 mm. Wide Sea-Salt Nuclei Deposited on 10 × 15 cm. Test Plate

FIG. 2.—Photomicrographs of Sea-Salt Nuclei Deposited on a Glass Strip 1 mm. Wide and on a 10 by 15-cm. Test Plate.

The slide areas photographed were selected to show the approximate range of nuclei size presented on the slides. Nuclei are shown as crystalline masses at low relative humidity and as liquid hemispheres at a higher relative humidity. (The outer edge of the dark ring marks the surface of the hemispheric drops. See photomicrographs of other sea-salt nuclei in Fig. 9.)

The absence of the larger nuclei on the smaller slide is attributed to the comparative sparsity of these nuclei in the air and to the short exposure time necessary for the small slide (Table I). The short exposure time for the 1 by 15-mm.

slide was necessary because of the danger that the high rate of deposition, if continued over a longer exposure time, might cause excessive coalescence of nuclei upon impact (due to the presence of too many nuclei on the surface) and thus modify the size distribution. Sampling sea salt with the smaller surface thus necessarily discriminates against the larger, sparsely distributed nuclei.[10] As suggested by the work of Langmuir[12], the fraction of the slides covered by the nuclei was kept below 10 per cent (Table I), thereby avoiding a significant amount of coalescence.

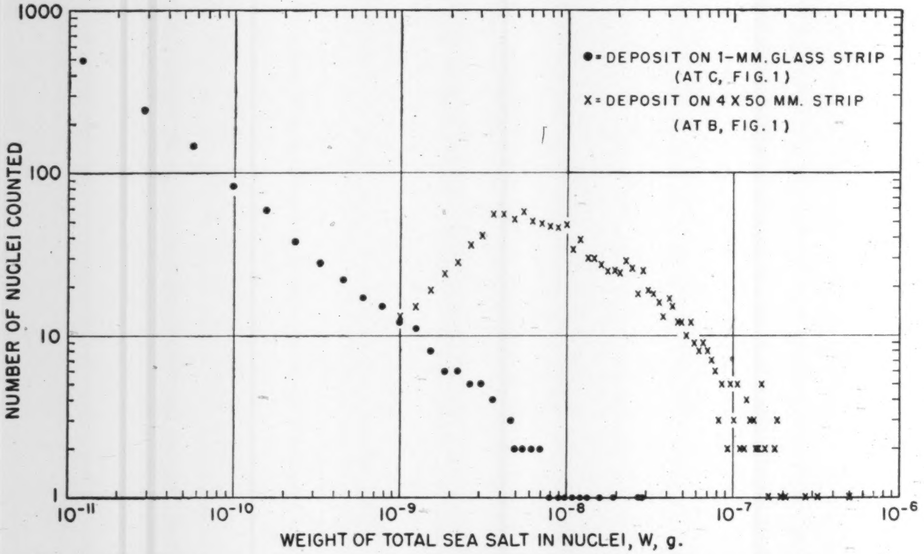


FIG. 3.—The Numbers and Weights of Sea-Salt Nuclei Counted and Measured on Two Surfaces of Different Dimensions Which Had Been Exposed Simultaneously at an Average Speed of Wind of 29 mph.

Table I also shows the rate of deposition of sea salt on the 4 by 50-mm. glass slide at specified distances from the edge of the plate. The last column in this table shows data concerning the salt deposited on the 1 by 15-mm. glass strip. Assuming that the 10 by 15-cm. plates generally used for corrosion tests are 1 mm. thick, the quantity of salt shown at the top of the last column represents the deposition rate on the edge of a plate when this edge is normal to the air stream. This result indicates that there would be about 6 times as much salt deposited on this edge when the *edge* is held normal to the stream as is deposited near this edge when the *face* is normal to the stream (Fig. 6). The deposition rate upon the edge held normal to the wind would be about 10 times the rate on the middle of the face of the plate held normal to the wind. This result indicates the importance of wind direction in effecting the rate of deposition of aerosols upon fixed surfaces such as test plates in exposure racks. This excess of deposition of sea salt on the plate edge may explain, in part, the excess of chlorides found by Copson[2] in the rust from the edges of steel plates exposed at Block Island and it may also explain the excessive "edge corrosion" on these plates.

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[10] This disadvantage of the sampling method may be eliminated by the use of sampling strips of several different widths. As indicated by the work of Langmuir and Blodgett[11] few of the more numerous small nuclei are deposited on wider strips ("ribbons") so that these strips may be exposed long enough to sample the large less numerous nuclei. If several different widths of sampling strips had been used, it is probable that the points indicating the weight and corrected number distribution would have fallen near a line such as the dashed line connecting the two groups of observed points on Fig. 5.

[11] Irving Langmuir and K. B. Blodgett, "A Mathematical Investigation of Water Droplet Trajectories," General Electric Research Laboratory Report, A.T.S.C. Contract W-33-038-AC-9151, pp. 1–47 (1945).

[12] Irving Langmuir, "Super-Cooled Water Droplets in Rising Currents of Cold Saturated Air," General Electric Research Laboratory Report A.T.S.C. Contract N-33-106-sc-65. Precipitation static studies, pp. 1–91 (1944).

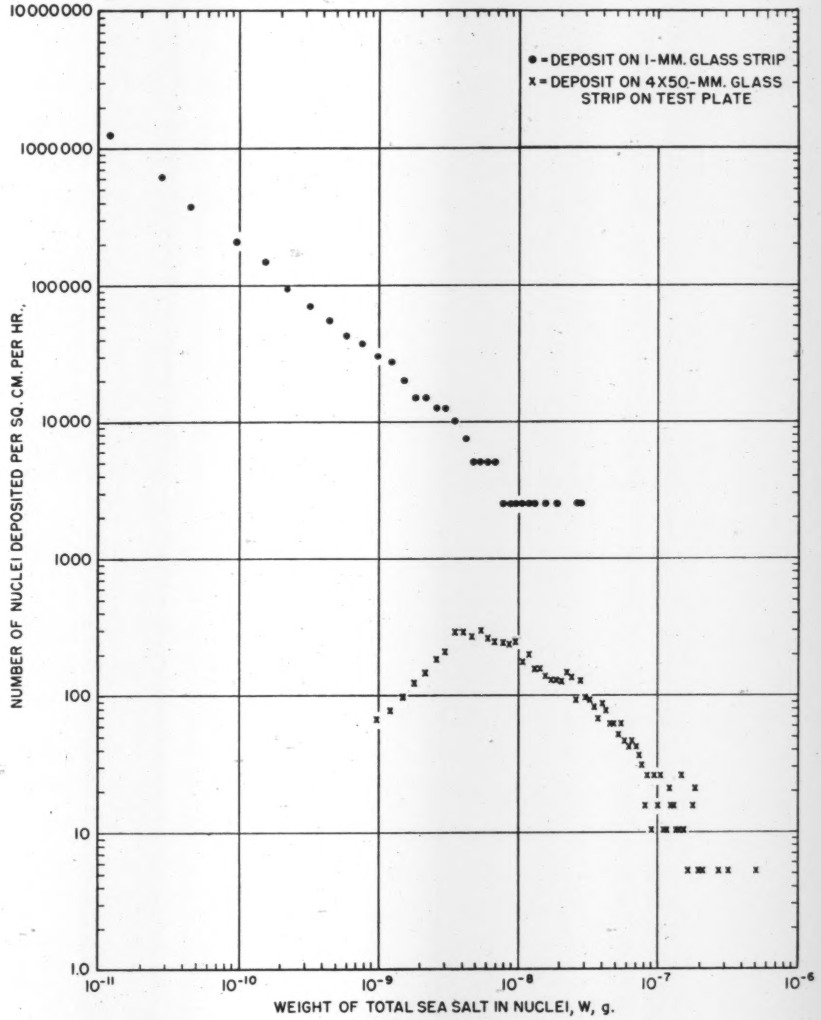

FIG. 4.—The Rates of Deposition of Sea-Salt Nuclei of Various Weights Upon a 1 by 15-mm. Glass Strip and a 4 by 50-mm. Glass Slide Which Was Inset on a 10 by 15-cm. Test Plate.

Figure 1 shows the mounting of these glass sampling surfaces and Table I gives further information about the conditions of exposure, etc.

Figure 7 shows the maximum variations, with wind speed and location, in the weights and numbers of sea-salt nuclei which the author has sampled in oceanic air by the impact method. Figure 8 shows the vertical distribution of nuclei in the sub-cloud layer of marine air during moderate winds.[13] This figure indicates an extensive vertical dispersion of nuclei, some of which are as large as those which were deposited upon the 10 by 15-cm. test plate. It should be noted that these nuclei (Figs. 7 and 8) were sampled at widely varying relative humidities. There seems to be little doubt that many of these nuclei would also impinge upon exposed steel test plates or other steel structures at these humidities and at appropriate wind speeds.

The purpose of Figs. 7 and 8 is to show something of the variability of the weight and number of sea-salt nuclei present in the lower atmosphere over and near the sea. Increases in nuclei sizes with increasing relative humidity, due to condensation of water vapor upon hygroscopic salts, will cause increases in the rate of deposition of the nuclei, as well as changes in the concentration of the salts in solution. Table II shows the computed sizes of salt nuclei as crystalline spheres and as liquid spheres at

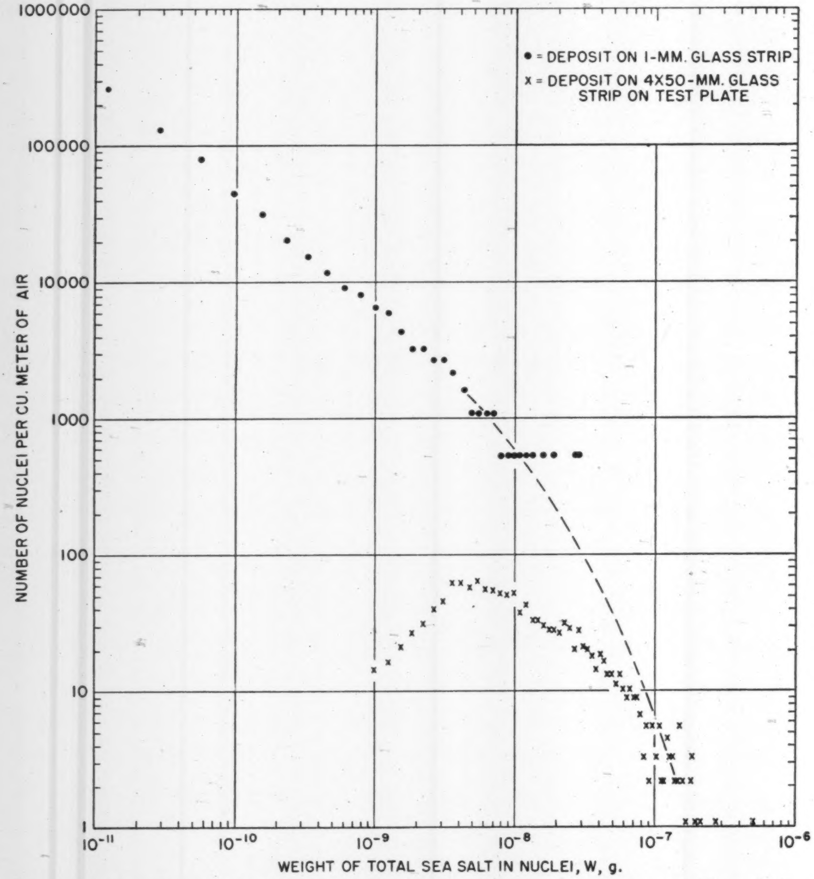

FIG. 5.—The Number and the Weight of Sea-Salt Nuclei per Cubic Meter of Air as Indicated by the Deposition on a Glass Strip 1 mm. Wide and on a 1- by 15-cm. Test Plate.[10]

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[13] Improved sampling techniques have recently indicated the presence of larger nuclei in the air. Thus in Figs. 7 and 8, it is possible that the points indicating weight distribution should be extended from one to two orders of magnitude in the direction of greater weights of salt.

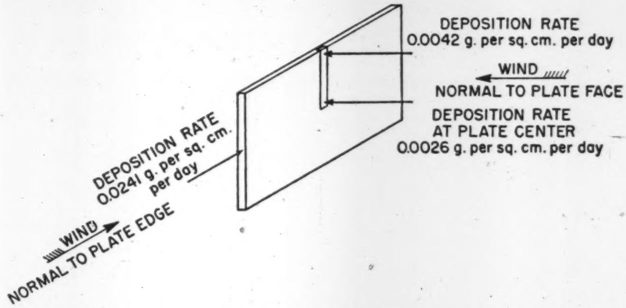

DEPOSITION RATE
0.0042 g. per sq. cm. per day
WIND
NORMAL TO PLATE FACE
DEPOSITION RATE
AT PLATE CENTER
0.0026 g. per sq. cm. per day
DEPOSITION RATE
0.0241 g. per sq. cm.
per day
WIND
NORMAL TO PLATE EDGE

FIG. 6.—An Illustration of the Effects of Relative Orientation of the Plate Surface and Wind Flow Upon the Rate of Deposition of Airborne Sea Salt. Wind Speed 12.9 meters per sec.

See Table I for further information about the sampling conditions.

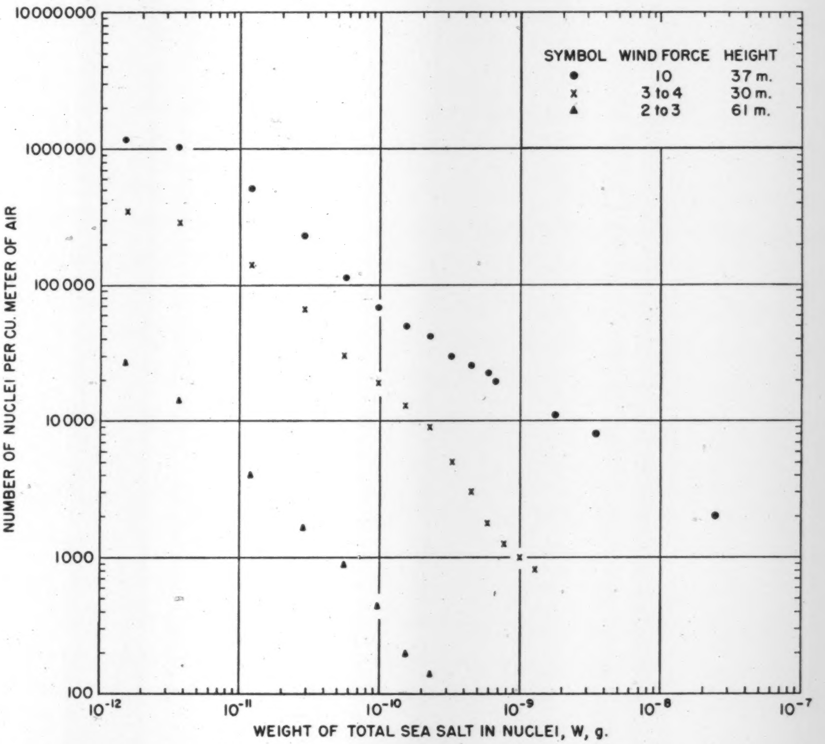


FIG. 7.—The Range of Weights and Numbers of Sea-Salt Nuclei Which Have Been Sampled on 1 by 15-mm. Glass Slides in Marine Air near the Sea Surface.[a]

[a] Some of the symbols used in the table are defined as follows:

T_d = dry bulb temperature of the air,
T_w = wet bulb temperature of the air,
R.H. = relative humidity, and
P_{mb} = atmospheric pressure in millibars.

Symbol	Date	Location	Position		T_d	T_w	R.H.	P_{mb}	Sampling speed, meters per sec.	Wind Direction
			Lat.(N)	Long. (W)						
●	September 22, 1948	Pompano, Fla.	26° 16′	80° 05′	27.0	24.7	83	997	28	ESE
×	November 26, 1948	Bermuda	32° 23′	64° 55′	21.2	15.3	43	1015	27	N
▲	August 22, 1949	Woods Hole, Mass.	41° 19′	70° 39′	20.1	17.6	78	1020	31	SW

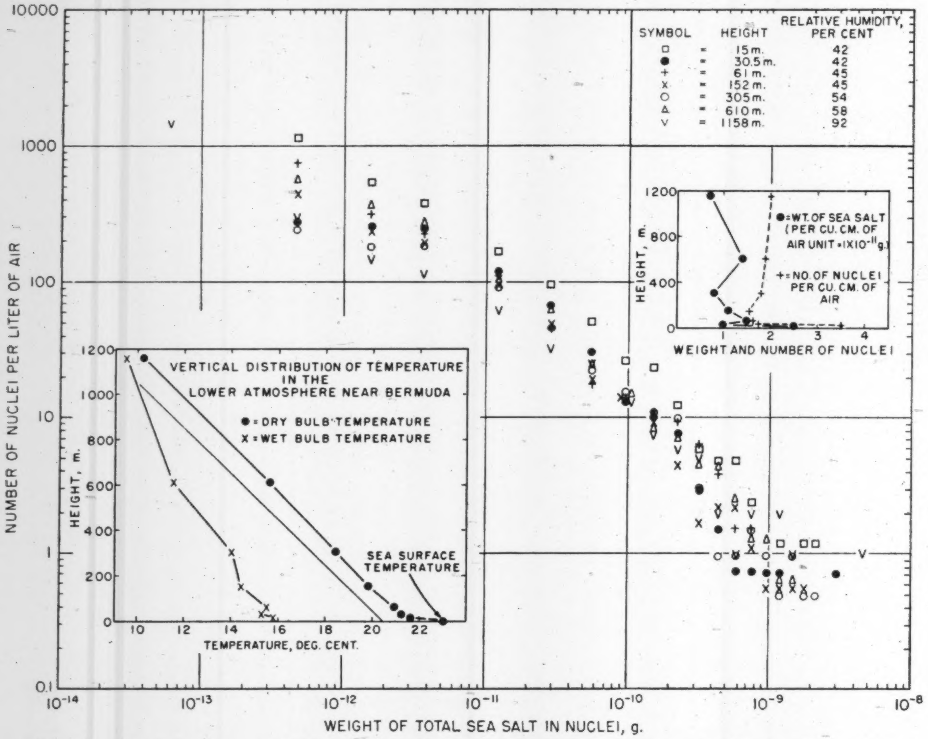


FIG. 8.—The Numbers and Weights of Sea-Salt Nuclei Which Were Sampled in the Lower Atmosphere Over the Sea Near Bermuda.

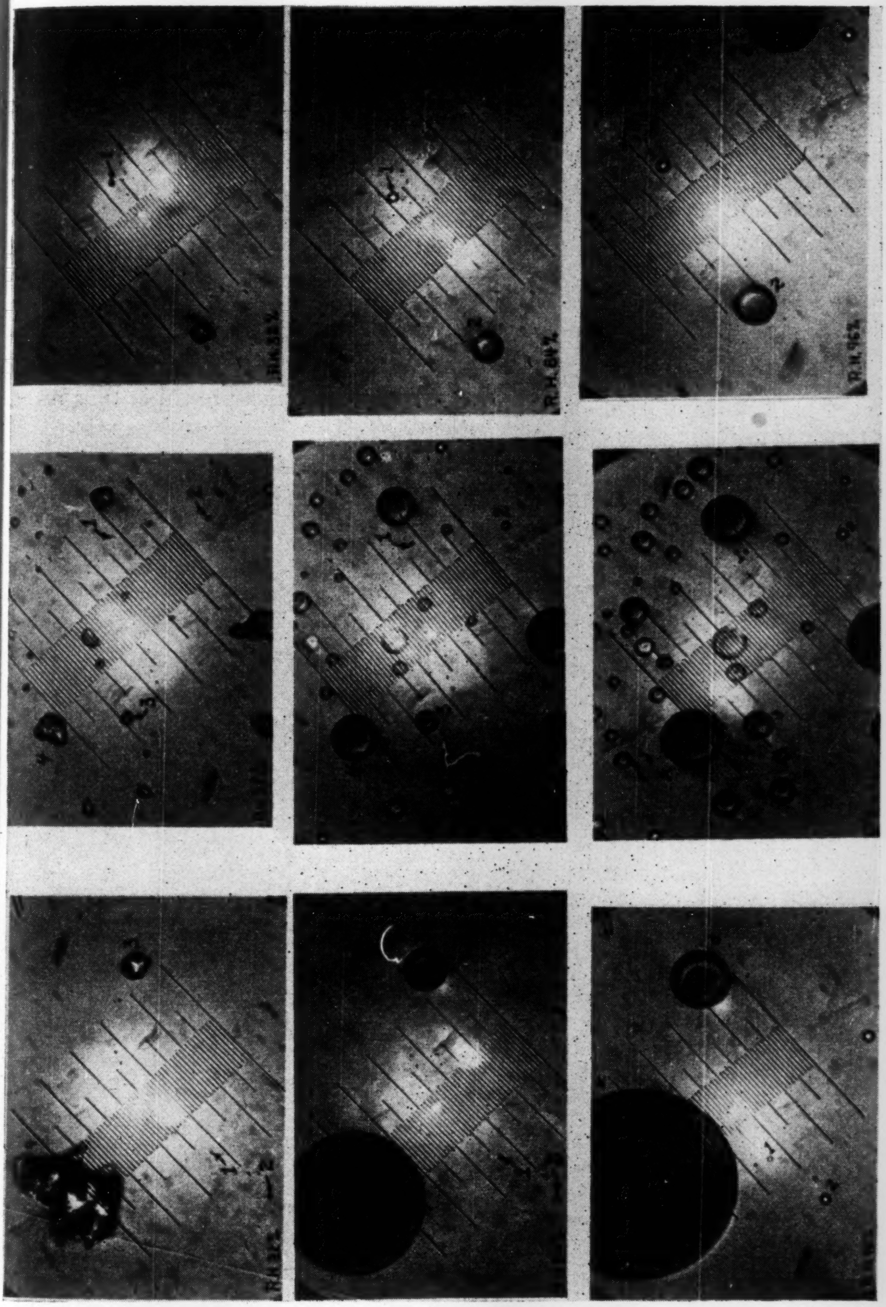
TABLE II.—COMPUTED SIZES OF SALT NUCLEI.

Weight of Total Sea Salt in Nucleus, g.	Nucleus Radius as Crystalline Sphere,[a] μ	Radius as Liquid Sphere at 80 per cent Relative Humidity, μ	Radius as Liquid Sphere at 95 per cent Relative Humidity, μ
1.5×10^{-12}	0.55	1.1	1.7
1.2×10^{-11}	1.1	2.0	3.2
1.5×10^{-10}	2.6	4.7	7.5
1.2×10^{-9}	5.15	9.4	15.1
1.2×10^{-8}	10.95	20.1	32.2
1.2×10^{-7}	24	44	70
5×10^{-7}	38	70	112

[a] The relative humidity at which the salt nuclei become crystalline seems uncertain. This is discussed in a paper by J. S. Owens entitled "Condensation of Water from the Air Upon Hygroscopic Crystals," *Proceedings*, Royal Soc., London (A), Vol. 110, pp. 738–752 (1926), in which he observed saturation at a relative humidity near 70 per cent. However, H. Dessens in his paper entitled "Brume et Noyaux de Condensation," *Annales de Geophysique*, Vol. 3, pp. 68–86 (1947), has indicated that some crystalline nuclei taken from the atmosphere remain liquid at much lower relative humidity values.

higher relative humidities. Figure 9 shows that concentrated sea water and hygroscopic nuclei from the air change from crystalline masses to liquid hemispheres and increase in size with increasing relative humidity. It is assumed that they would also change in this manner in the free air as the relative humidity changed.

The methods used to make the above measurements and computations about atmospheric sea salt[8] should be readily applicable to measurements concerning sea salt deposition on other objects or structures exposed to marine winds. The importance of the shape and size of surfaces, and of the wind speed in modifying the rate of impact deposition of aerosols,[11] makes these factors of primary interest to those concerned with aerosol deposition on steel or other structures. The present author's experience in sampling concentrated sea-water aerosols in the atmosphere indicates that the roughness of the surface exposed to the air



Concentrated Sea Water Nuclei. Atmospheric Nuclei, Alt. 610 meters, Bermuda. Atmospheric Nuclei, Alt. 15 meters, Woods Hole.

FIG. 9.—Photomicrographs of Changes in the Size of Concentrated Sea Water Nuclei and of Atmospheric Nuclei as the Relative Humidity Over Them is Changed. (Scale: one division = 2.2μ.)

stream is also an important factor in modifying the rate of salt accumulation at localized areas. For instance, a minute protuberance on a cylindrical surface might be expected to receive a greater proportion of the impinging aerosols than would adjacent smoother surfaces.

The measurements given in this paper indicate the importance of wind in causing aerosols to impinge upon test plates or other surfaces. The position and the amount of such impingement on a fixed test plate at a given location will depend, to a large extent, upon wind speed and direction. The impinging aerosols will usually continue to accumulate until rain washes them off. Accumulations of these impinged aerosols on surfaces exposed to the wind but sheltered from rain might be expected to bring about greater corrosion on these surfaces. An excess of corrosion on steel surfaces exposed to winds but sheltered from rains has been found by Larrabee[4] and by Pilling and Wesley.[7] In a given region the stronger prevailing marine winds should cause greater deposition of aerosols on certain surfaces of fixed test plates. It is suggested that the excessive corrosion found by Copson[2] on the west side of plates at Block Island may be due to an excess of sea salt deposited on that side by the westerly winds which prevail in that region.[14]

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[14] U. S. Dept. of Agriculture, Weather Bureau, "Atlas of Climatic Charts of the Oceans," Washington, D. C. (1938).

DISCUSSION

Mr. H. R. Copson[1] (*presented in written form*).—Mr. Woodcock has presented some fundamental data which unquestionably are of importance in atmospheric corrosion. He is to be commended for obtaining quantitative measurements of the amount and distribution of air-borne deposits. It is to be hoped that the data will be extended to include other angles of wind impingement besides normal to the specimen, and that other conditions of atmospheric corrosion tests will be simulated more closely.

TABLE III.—EFFECT OF NEARNESS TO OCEAN.[2]
Specimens exposed at Kure Beach, N. C., for 1.39 yrs.

Steel	Weight Loss, g. per sq. dm.	
	800 ft. from Ocean	80 ft. from Ocean
No. 1	9.45	gone
No. 11	4.33	gone
No. 6	4.33	16.90
No. 39	3.16	5.03

TABLE IV.—RELATIVE CORROSION RATES OF WIRE AND SHEET METAL.[3,4]

Values of the average thickness of the corroded layer expressed in inches per year × 10^{-4}. Duration of test, 1 yr. Corrosion rates of wires determined by the electrical resistance method. Corrosion rates of sheet determined by weight losses.

Type of Atmosphere	Test Site	70-30 Nickel-Copper		Nickel		80-20 Nickel-Chromium	
		Wire	Sheet	Wire	Sheet	Wire	Sheet
Rural	Cardington	1.72	0.57	1.68	0.45	0.78	0.17
Industrial	Bourneville	3.43	1.24	2.74	0.96	1.33	0.46
	Wakefield	4.78	1.34	5.60	2.18	1.75	0.62
	Birmingham	6.56	2.43	5.64	2.30	3.11	0.85
Marine	Southport	3.94	1.13	3.25	1.11	1.37	0.26
Average	All	4.08	1.34	3.78	1.40	1.67	0.47

That the amount of sea salt deposited on steel specimens is important is shown by the data in Table III.[2] Identical specimens were exposed on racks located 80 ft. and 800 ft. from the ocean. Specimens nearer the ocean corroded much faster than those further away, undoubtedly because more sea salt was deposited on them.

Mr. Woodcock's observations probably explain one aspect of atmospheric corrosion which he has not mentioned. There seems to be a general rule that the rate of corrosion of wire specimens is greater than that of sheet. The magnitude of this effect is indicated by Hudson's data in Table IV.[3,4] The only explanation offered hitherto has been that layers of corrosion product form imperfectly on wire of small diameter, but this explanation hardly seems tenable in the case of passive films. The data in the paper indicate that the rate of deposition of sea salt on wire would be much greater than on sheet. Presumably other kinds of air-borne pollution would behave similarly to sea salt.

Reference is made in the paper to edge corrosion of steel specimens at Block Island. In this connection it would seem that the final resting place of the salt would be more important than the place it was deposited. It is instructive to note the amount of sea salt deposition reported in the paper. On a 10 by 15-cm. specimen the average figure was 3 mg. per sq. cm. per day. At this rate the accumulation in 3 yr. would be 500 g. At Block Island the 12 g. of rust on both sides of a 3-yr. old 10 by 15-cm. specimen analyzed only 0.04 per cent chloride.[5] This is equivalent to about 10 mg. of sea salt on the specimen. Of course the bulk of the salt washed away. It seems impossible that this washing action could have other than a major effect on the distribution of the salts.

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[1] Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

[2] H. R. Copson, "Factors of Importance in the Atmospheric Corrosion Testing of Low Alloy Steels," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, 581–609 (1948).

[3] J. C. Hudson, "Third Report of the Atmospheric Corrosion Research Committee," *Transactions*, Faraday Soc., Vol. 25, pp. 177–252 (1929).

[4] W. A. Wesley, "The Behavior of Nickel and Monel in Outdoor Atmospheres," Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals, p. 23, Pittsburgh Spring Meeting of Am. Soc. Testing Mats. (1946). (Symposium issued as separate publication *STP No. 67*.)

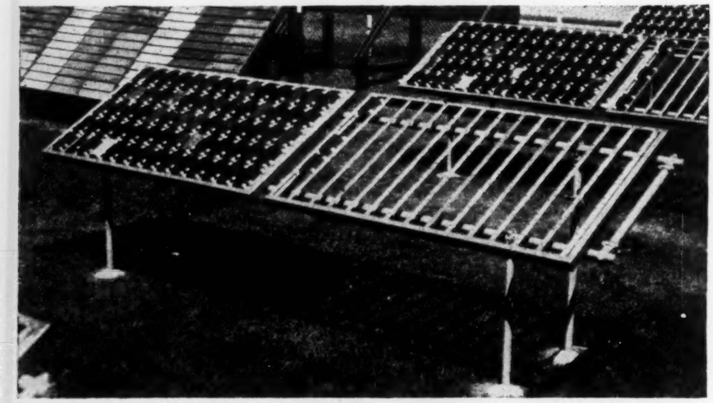

FIG. 10.—Manner of Exposure of 4 by 6 in. Steel Specimens.[5]

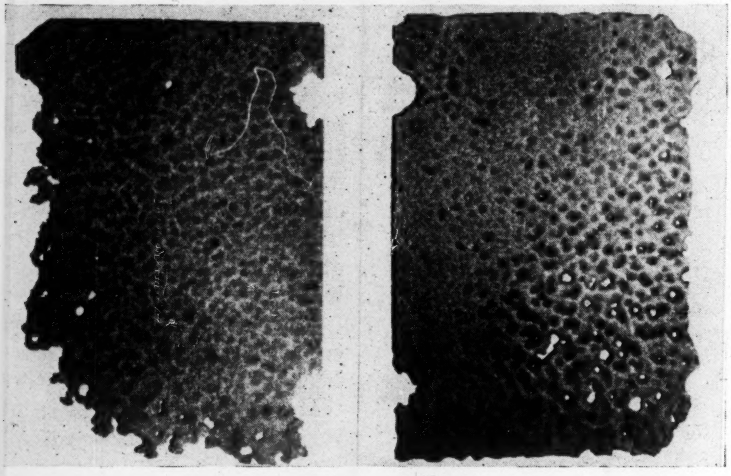

FIG. 11.—Typical Edge Effects at Block Island After 3 yr.[5]

It might be helpful to see exactly what the edge corrosion looks like. Figure 10 shows the exposure conditions.[5] The 4 by 6-in. specimens were mounted closely side by side and were exposed at an angle of 30 deg. from the horizontal facing south. They were held in position by porcelain insulation.

Figure 11 illustrates the edge corrosion. The left-hand picture shows the skyward surface of one specimen and the right-hand picture shows the groundward surface of a second specimen. Two circular holes were drilled in each specimen for identification purposes, but the rest of the perforations were formed by corrosion.

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[5] H. R. Copson, "A Theory of the Mechanism of Rusting of Low Alloy Steels in the Atmosphere," *Proceedings*, Am. Soc. Testing Mats., Vol. 45, pp. 554–590 (1945).

The corrosion is severe at the left or western edge, at the groundward edge, and also under the insulation at the right edge. On the skyward surface the deepest pits were within 1 in. of the corroded edge, while on the groundward surface pits were still deep 2 or 3 in. from the corroded edge. This pattern of corrosion was repeated over and over for all positions on the rack.

In the paper it is pointed out that the prevailing winds are westerly. If this were the important factor, it would be expected that the edge corrosion would be more severe at the western portions of the racks than at the eastern portions, and such is not the case. It is difficult to see how the big difference between the upper and lower edges, between the skyward and groundward surfaces, and the severe attack near some insulators can be explained by the position at which sea salt deposits.

Actually it has been found that at Block Island on rainy days the prevailing wind is not from the west but instead is from the east. All the localized attack can be explained by the washing action of the easterly rain. Soluble salts are washed from the upper right corner toward the lower left corner. The upper edge is thoroughly washed and does not corrode very much. Washing is poor near the insulators and corrosion is rapid there. The washing is not as effective on the groundward side and the attack is not as localized on this side.

In an attempt to learn more about the

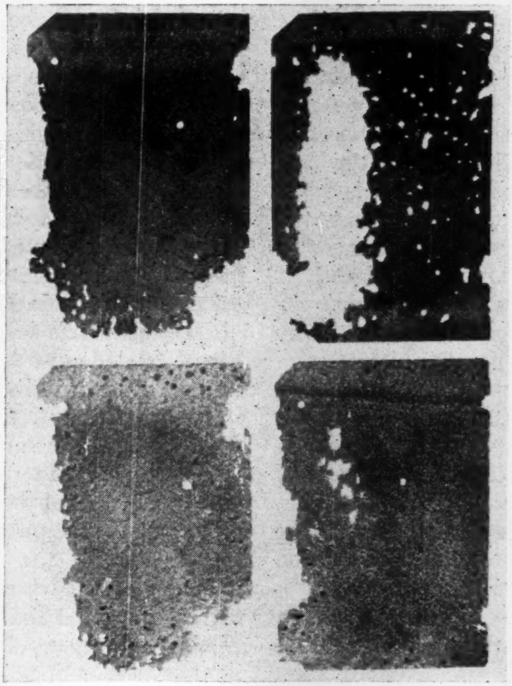

FIG. 12.—Effect of Exposure Direction.

Upper two specimens one steel, lower two another steel. Right-hand specimen exposed facing east, left-hand specimens facing south.[2]

edge corrosion, a few specimens were exposed facing east instead of south. Figure 12 shows the result. The upper two specimens are of one steel and the lower two another. The right-hand specimens were exposed facing east and the left-hand specimens facing south. The pattern of corrosion on the south specimens was about the same as before. On the east specimens the severest corrosion occurred on one side of the center line but back from the edge. During the period of exposure, 53 per cent of the total rain fell when winds were from northerly directions. Since the corrosion was more severe toward the side opposite the predominant rain direction, there is some correlation in this respect. However the correlation is not good with respect to greater salt deposition at the edges.

The main purpose of these observations is to point out that there are many factors involved in the rate of rusting of steel in the atmosphere. Mr. Woodcock has emphasized one factor, but this should be used to supplement rather than to exclude other factors. The manner of exposure, shelter, washing action of rain, alloy content of the steel, the protective character of the rust, and the duration of exposure are all important.

MR. A. H. WOODCOCK (*author*).—Mr. Copson's comment about the greater corrosion rate of wire compared to sheet specimens may serve to emphasize the importance of the shape and size of surfaces upon the rate of impact deposition of aerosols (see p. 1158 of my paper). The roughness of the surfaces and their orientation in the air stream are also important in modifying aerosol deposition rates.

I would not expect that westerly winds at Block Island would cause a greater edge corrosion at the western portions of the racks than at the eastern end as suggested by Mr. Copson. There should be little difference in either the aerosol content of the air or in the wind speed at the two ends of the racks.

It should be reemphasized that the rate of impact deposition of aerosols upon test plates in fixed exposure racks will probably prove to be greatly dependent upon the speed and direction of air flow in the immediate vicinity of the plates. Rates of impingement of aerosols on these plates could be determined by the insertion of recessed removable glass slides at critical areas. A thermister anemometer[6] might be useful in exploring the rates of air flow close to the plate surfaces during winds of various speeds from various directions. When measurements such as these are available we should be, in my opinion, better able to judge their importance in understanding irregular corrosion rates such as those on the plates shown in Figs. 11 and 12.

Copson's plates suggest to me that at Block Island most of the aerosols are deposited during winds from the southwesterly quadrant. Such winds should deposit about the same number of particles on the western and bottom (or southern) edges of the slides tilted south (Fig. 11). On the plates tilted east (Fig. 12, right side) the greatest corrosion seems to be on back southwest side. The corrosion around the supporting insulators may be due to downward deflection of the air stream by the projecting insulators. This deflection would produce increased local deposition of aerosols due to the increased angular acceleration within the air stream near the insulators.

The validity of these suggestions concerning a relationship between corrosion patterns and the patterns of impingement of aerosols can probably be indicated by a study of the patterns of impingement on fixed test plates in exposure racks.

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[6] Wayne B. Hales, "Thermisters as Instruments of Thermometry and Anemometry, *Bulletin*, Am. Meteorological Soc., Vol. 29, pp. 494–499 (1948).

MR. F. L. LAQUE.[7]—I would like to ask Mr. Woodcock a question as to his opinion of the practicability of measuring the amount of salt particles, if you can call them such, in an air stream by absorbing the passing stream or a portion of it in an absorption train of some sort. This procedure would not necessarily measure how many salt particles might be deposited from the stream on a particular surface, but it might serve reasonably well to show the relative number of salt particles available for deposition on surfaces such as test specimens exposed 800 ft. from the ocean as compared to similar specimens exposed 80 ft. from the ocean. We have available some apparatus which could be used along the lines indicated and I would, therefore, like to have Mr. Woodcock's opinion as to whether such a technique would give us a reliable measure of the availability of salt particles for deposition on test pieces at different wind velocities and at different distances from the ocean.

MR. WOODCOCK (*author*).—Filtering methods have been used to measure airborne sea salt.[8] May[9] has shown that errors occur if the velocity of flow within the air intake orifice of the filter is not maintained equal to that of the ambient air. However this orifice error should prove to be insignificant for your work, since the quantity of sea salt in the air over the sea varies from 10 to 100 times during the average range of winds.

MR. SAM TOUR.[10]—In connection with the impingement collection on glass plates, the author did not mention the surface preparation of the glass plates. How were the surfaces prepared to receive a particle when it did impinge, and to hold it there?

In connection with dust sampling, in general, the problem is two-fold, one, getting the particle to impinge and, second, getting the particle to stay there, once it has impinged. It would appear, from the illustrations, that the particles had a tendency to wet the glass.

In connection with sampling for total salt content, I think the much simpler answer to Mr. LaQue's question would be electrostatic sampling of a sizable number of cubic feet of air to get the total salt content very rapidly.

MR. WOODCOCK (*author*).—The glass slides were coated with Dri-film[11] in the manner suggested by Langmuir.[12] The Dri-film coating on the slides produces a hydrophobic surface on which water droplets become hemispheric, making it possible to determine the volume of the droplets from diameter measurements.

The high solubility of some of the sea salts probably causes the sea-salt nuclei to remain wet at relative humidities as low as 33 per cent. For this reason it is assumed that the crystalline nuclei will readily adhere to the glass slides at humidities above 33 per cent. I have seen no evidence that the nuclei ever bounce off the glass slides. Sedimentation sampling of these nuclei gives the same distribution of weight as does the impact sampling.

MR. TOUR.—On the question of air sampling, and particulate matter carried in air streams, there are some interesting things. Particles below a given size normally do not tend to drop out, but tend to move upwardly, when the air is moving up, and will move downwardly when the air is moving down. Without a wind of any velocity present air tends to move upward and downward. It does not stay static. With no cross wind present, the underside of the plates exposed on a rack can be wet and impinged upon. Matter reaches the underside of the plate, with no wind present and apparently a static atmosphere, due to the up and down currents which result regularly from the difference in temperature between the ground and the air above it.

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[7] In Charge of Corrosion Engineering Section, Development and Research Div., The International Nickel Co., Inc., New York, N. Y.

[8] W. C. Jacobs, "Apparatus for Determining the Salt Content of the Air," *Bulletin*, Am. Met. Soc., Vol. 20, pp. 38–42 (1939).

[9] K. R. May, "The Cascade Impactor. An Instrument for Sampling Coarse Aerosols," *Journal of Scientific Instruments*, Vol. 22, pp. 187–195 (1945).

[10] Chairman of the Board, Sam Tour and Co., Inc., New York, N. Y.

[11] Dri-film No. 9987, made by the General Electric Co., Schenectady, N. Y.

[12] Irving Langmuir, "Super-Cooled Water Droplets in Rising Currents of Cold Saturated Air," General Electric Research Laboratory Report, A.T.S.C. Contract W-33-038-AC-9151, pp. 1–47 (1944).

MR. WOODCOCK (*author*).—At very low rates of air flow, the collection of aerosols by impingement should approach the rate of collection by sedimentation. The rate at which salt nuclei fall out of relatively still air (for example, g. per sq. cm. per day) has been found to be only about one one-thousandth of the rate at which these nuclei impinge upon a 1-mm. wide glass slide in the same air moving at 10 m. per sec.[13] From these measurements, I would estimate that the rate of sedimentation or falling out of salt nuclei on 10 by 15-cm. test plates in still air would be about one one-hundredth of the average rate of impingement on a plate held normal to a wind of 10 m. per sec.

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[13] These data on the relative rates of impingement and sedimentation of sea-salt nuclei will be published at a later date.

REPRODUCIBILITY OF RESULTS OF TENSION TESTS ON SPECIMENS PREPARED FROM CAST STEEL COUPONS*[1]

By H. A. Schwartz[2] and W. K. Bock[2]

Synopsis

Data are recorded showing the degree of reproducibility of the usual static tensile properties of grade "B" steel as determined on test coupons of several designs.

The results of single tests or the average of a small number of tests is shown not to be an adequate measure of the mechanical properties of a given steel cast in a given form of coupon.

In connection with specification writing and other matters involving the discussion of the tensile properties of steel, one experiences the need of judging how accurately a single test, or the average of a few tests, may be expected to represent the true average values of tensile properties of a particular heat of steel.

The original purpose of the present investigation was to determine the uniformity of results to be expected from tests made by the usual twin-keel block specimens. Very early it became obvious that it would be desirable to see if the uniformity of results was in any way related to the type of test coupon.

The types of coupon design selected for study were the keel block (two types—twin and multiple), the four-leaf clover type, and a large block feeder carrying eleven vertical test bars on its sides. The coupon designs are shown in Figs. 1, 2, 3, and 4.

Test Methods

The plant which made the horizontal keel-block coupons was in the habit of making them by casting a plate 29 in. square by about 5 in. thick, to whose lower surface several dry sand cores are applied to form the legs. The slab will hold approximately 50 coupons.

It was originally planned to make all of the specimens in green sand. This did not prove feasible from an operating viewpoint, and the vertical keel block (Fig. 3) was finally poured in green sand, the cloverleaf coupon (Fig. 4) in dry sand and the horizontal keel block (Fig. 2) and the twin-keel block (Fig. 1) with the legs in dry sand and the feeders in green sand. When this part of the work was practically completed, the question was raised whether the horizontal keel block (Fig. 2) gave as uniform results as the twin-keel block. For this purpose two more horizontal keel blocks were cast and an appropriate number of twin keel block feeders were also cast.

Two heats of steel are involved—one covering the coupon designs of Figs. 2, 3, and 4, the other covering designs of

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] This paper sets forth the results of research conducted by the National Malleable and Steel Castings Co. and supported by the Steel Founders' Soc. of America.

[2] Manager of Research and Research Engineer, respectively, National Malleable and Steel Castings Co., Cleveland, Ohio.

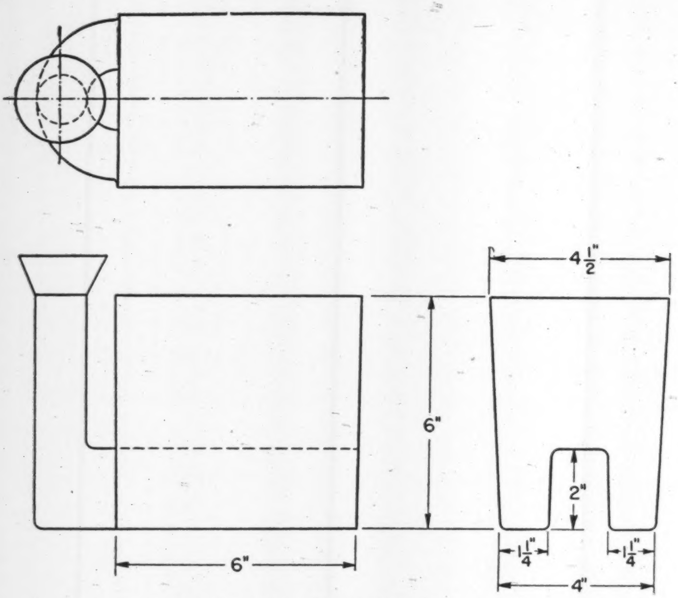

FIG. 1.—Twin Keel Block Coupon.

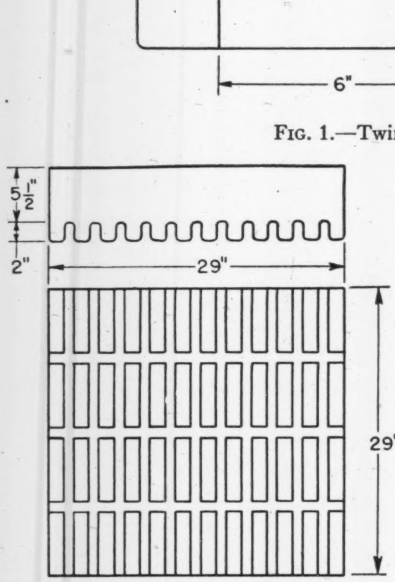

FIG. 2.—Horizontal Keel Block Coupon.

Figs. 1 and 2. The horizontal keel block design (Fig. 2) was produced in both heats. The compositions of the heats were as shown in Table I. Both heats were deoxidized with 3 lb. of calcium silicon plus 4 lb. of carbortam per ton.

TABLE I.—COMPOSITION OF STEELS.

	First Heat	Second Heat
Carbon, per cent	0.27	0.30
Manganese, per cent	0.53	0.55
Silicon, per cent	0.57	0.55
Sulfur, per cent	0.023	0.025
Phosphorus, per cent	0.026	0.021
Chromium, per cent	0.045	0.08
Vanadium, per cent	none	none
Nickel, per cent	0.08	0.08
Copper, per cent	0.24	0.15
Molybdenum, per cent	none	0.02

Somewhat over 50 specimens of each type were cast from a single heat. It had been the intention to mark each coupon so that pouring sequence and position in the heat treating furnace could be accurately reported. Under shop conditions this proved impossible. However, the test bars from each particular type of coupon were placed close together in a small commercial heat treating furnace, so that it is known that all of the bars from a coupon design were heat treated alike.

The heat treatment for each heat on all test bars was conducted at the same time in the same furnace. For the first heat involving the horizontal keel block, the cloverleaf, and the vertical keel

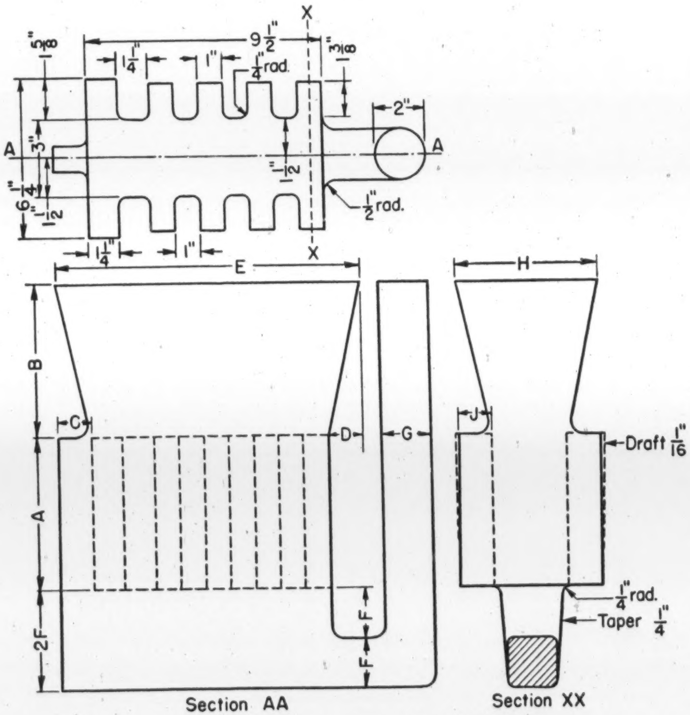
9 1/2"
1 5/8"
1 3/8"
1 1/4"
1"
1/4" rad.
2"
X
A
3"
1/2"
6 1/4"
1 1/2"
1/2" rad.
E
H
B
C
D
G
J
Draft 1/16"
A
1/4" rad.
Taper 1/4"
F
2F
Section AA
Section XX

FIG. 3.—Vertical Keel Block Coupon.

A = 6 in.
B = 6 in.
C = 1 3/8 in.
D = 1 3/8 in.
E = 12 1/4 in.
F = 2 in.
G = 2 in.
H = 5 3/4 in.
J = 1 3/8 in.

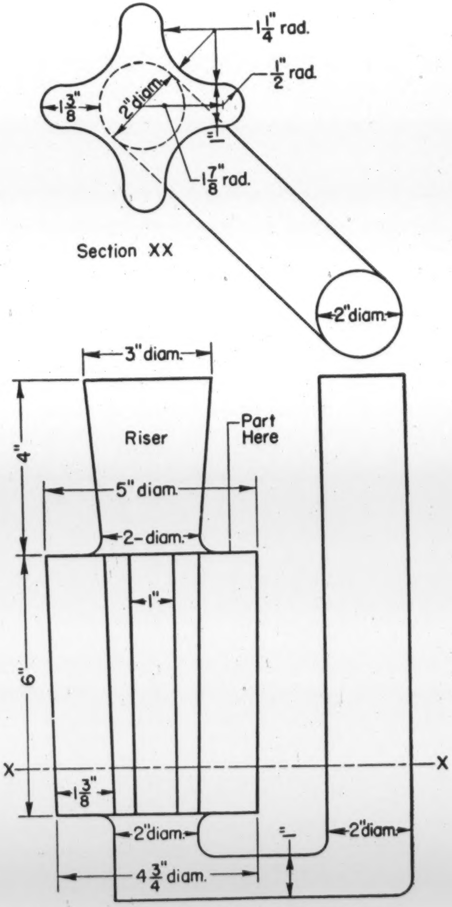
1 1/4" rad.
1/2" rad.
1 3/8"
2" diam.
1"
1 7/8" rad.
Section XX
2" diam.
3" diam.
Riser
Part Here
4"
5" diam.
2-diam.
1"
6"
X
X
1 3/8"
2" diam.
1"
2" diam.
4 3/4" diam.

FIG. 4.—Cloverleaf Coupon.

block, the heat treatment consisted of normalizing from 1690 F. (920 C.) after holding 2 hr. at temperature, and tempering for 2 hr. at 1160 F. (625 C.). For the second heat of steel the heat treatment was as follows: Normalized from 1700 F. (925 C.) after holding 2 hr. at temperature, and tempered 2 hr. at 1160 F. (625 C.).

The castings were made by the Sharon Works of the National Malleable and Steel Castings Co. from basic open-hearth steel, and the test specimens were machined at the Sabin Machine Co. in Cleveland. The specimens were of the shouldered form described in the Standard Methods of Tension Testing of Metallic Materials (E 8 – 46).[3] They were deliberately given a slight taper toward the center as permitted by the method. Steel analyses and the tension tests were made in the Research Laboratory of National Malleable and Steel Castings Co.

In a study requiring the comparing of various sizes and shapes of test coupons, there is always the possibility of obtaining defective test bars. For example, the plant that produced the coupons poured them from a 30-ton capacity ladle having a $1\frac{1}{4}$ in. diameter nozzle. The use of such pouring procedure resulted in obtaining some defective bars on the smaller coupon designs from entrapped air entering with the stream, etc., which would not be present if hand shank pouring had been used. The need for rejecting such test bars did not alter the accuracy of the conclusions for the following reasons:

1. Gross defects are easily detected during machining, or before testing, or by observing the broken fracture, and can be rejected from test results.

2. None of the test bars used in the study, which were only the sound bars, departed in properties by more than 3σ from the mean value.

TEST RESULTS

The individual values of the several static tensile properties constitute so large an accumulation of data, that it is believed that a tabulated presentation would be of but little interest. They can best be summarized by the usual methods of statistics.

Fifty points are not sufficient to justify the application of a χ^2 test to see whether the distribution is sufficiently well represented by a "normal" curve. Plotting suggests that the reduction of area distribution is strongly skewed toward high values. In accordance with most quality control practices, we have nevertheless contented ourselves, perhaps rashly, with the average and standard deviation as criteria of the universes. The mean values and standard deviations of the five lots of coupons are shown in Table II.

The likelihood that the differences observed in Table II have occurred as the result of chance only are shown in Table III.

The mechanics of the test as outlined by Rider[4] consists in setting up the null hypothesis that the two means in question come from the same normal population and then determining by means of the t-test the probability of obtaining as great a difference as that observed, based on the assumption made. Thus at a 5 per cent confidence level a probability greater than 5 per cent would not be sufficient evidence to discard the null hypothesis, and so it would not be judged that the means are significantly different.

When a difference is significant it means that two universes or lots cast from different patterns are not interchangeable. When the difference is marked "not significant," then the two groups or universes are indistinguishable

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[3] 1949 Book of A.S.T.M. Standards, Part 1, p. 1233.

[4] *Statistical Methods*, John Wiley and Sons, Inc., New York, N. Y.

TABLE II.—MEANS AND STANDARD DEVIATIONS.

Pattern	Sample Size	Yield Point, psi.		Tensile Strength, psi.		Elongation, per cent		Reduction of Area, per cent	
		Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation	Mean	Standard Deviation
Heat No. 1									
Clover leaf	26	46110	1144	79473	420	32.4	2.1	54.2	4.1
Horizontal keel block	40	43945	1164	77218	538	31.3	1.7	54.8	3.0
Vertical keel block	37	44641	846	79472	190	31.0	1.9	54.2	2.5
Heat No. 2									
Double keel block	50	44028	1126	78462	656	31.5	1.6	53.6	1.9
Horizontal keel block	46	43772	1316	78558	629	28.9	1.9	46.2	4.3

TABLE III.—SIGNIFICANCE OF DIFFERENCE OF MEANS.

Patterns Compared	Property	Probability[a]	Significance of Difference
Heat No. 1			
Horizontal keel block *versus* vertical keel block	yield point	<0.01	significant
	tensile strength	<0.01	significant
	elongation	$=0.02$	significant
	reduction of area	$=0.50$	not significant
Horizontal keel block *versus* vertical keel block	yield point	<0.01	significant
	tensile strength	<0.01	significant
	elongation	$0.40<P<0.50$	not significant
	reduction of area	$0.30<P<0.40$	not significant
Cloverleaf *versus* vertical keel block	yield point	<0.01	significant
	tensile strength	>0.90	not significant
	elongation	<0.01	significant
	reduction of area	1.00	not significant
Heat No. 2			
Double keel block *versus* horizontal keel block	yield point	$0.3<P<0.4$	not significant
	tensile strength	$P=0.5$	not significant
	elongation	$P>0.01$	significant
	reduction of area	$P>0.01$	significant

[a] Various signs and abbreviations in this column are explained as follows:

P … probability
$<$ … less than
$>$ … greater than
$=$ … approximately

TABLE IV.—PROBABILITY THAT THE DIFFERENCES OF SCATTER ARE DUE ONLY TO CHANCE.

Property	Probability[a]	Number of Universes	Odd Variance
Heat No. 1			
Yield point	$0.1>P>0.2$	2 universes	vertical keel block
Tensile strength	$0.01>P$	2 universes	vertical keel block
Elongation	$0.5>P>0.6$	same universe	....
Reduction of area	$0.02>P>0.05$	2 universes	cloverleaf
Heat No. 2			
Yield point	$P<0.05$	same universe	....
Tensile strength	$P<0.05$	same universe	....
Elongation	$P<0.05$	same universe	....
Reduction of area	$P>0.5$	different universes	....

[a] Various signs and abbreviations in this column are explained as follows:

P … probability
$<$ … less than
$>$ … greater than

and different only by chance. The probability 0.05 has been arbitrarily chosen as the "fiduciary limit," which if exceeded renders the difference insignificant. It is quite possible that before condemning a given design of coupon the comparison should be based on a much smaller limit.

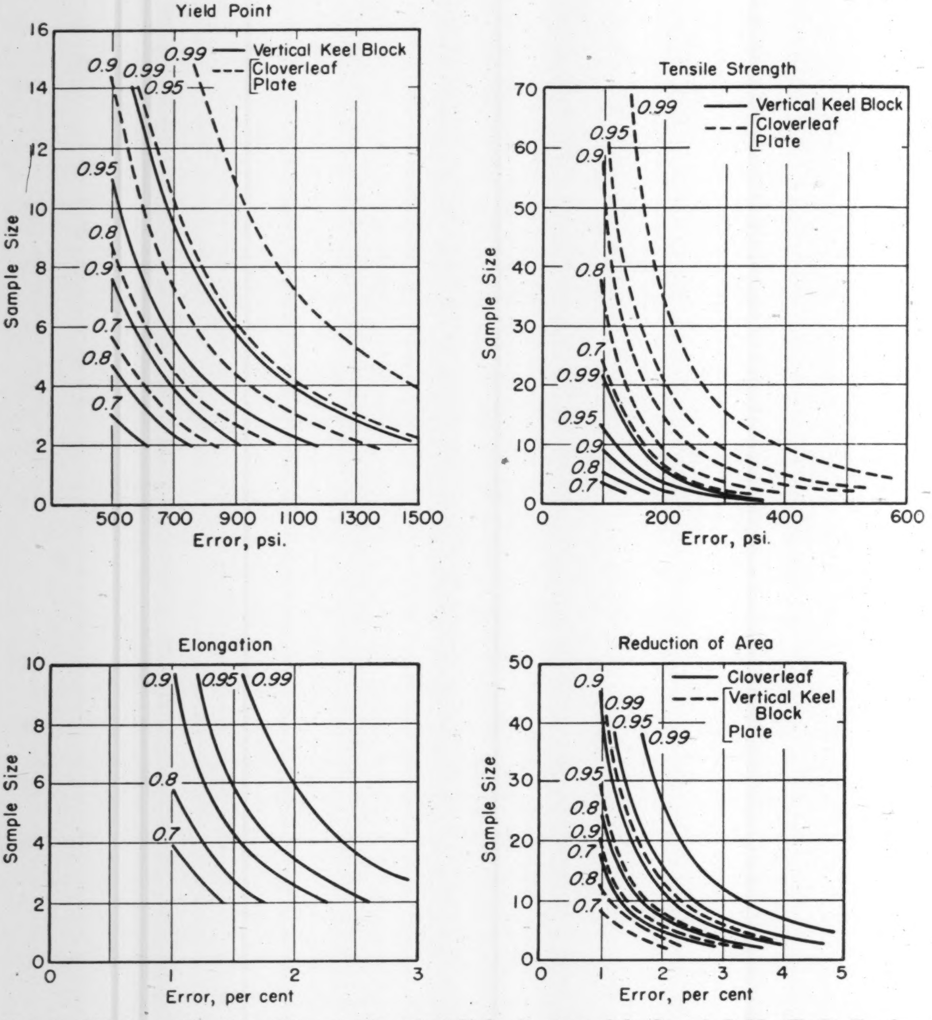

FIG. 5.—Minimum Number of Samples Required If the Average of the Sample Is Not To Be Further Than the Allowed Error from the True Average in 0.7, 0.8, 0.9, 0.95 and 0.99 Cases (7, 8, and 9 Chances in 10; 95 and 99 Chances in 100).

Rider[4] gives a method of checking the homogeneity of several variances based on differences between the variances and their weighted mean. The probabilities of obtaining these differences by chance are shown in Table IV. At a 5 per cent fiduciary limit the variances of yield point are not shown to be dif-

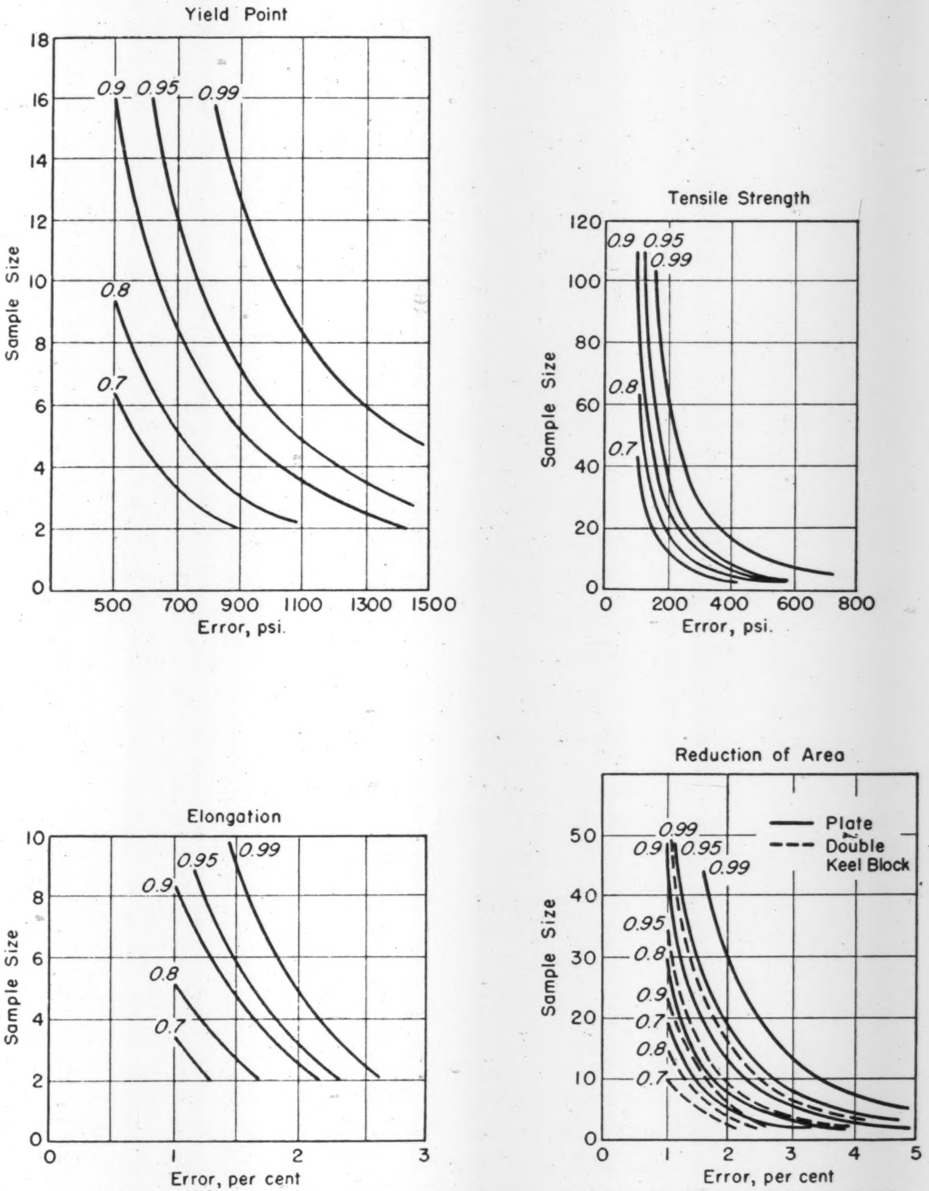

FIG. 6.—Minimum Number of Samples Required If the Average of the Sample Is Not To Be Further Than the Allowed Error from the True Average in 0.7, 0.8, 0.9, 0.95 and 0.99 Cases (7, 8, and 9 Chances in 10; 95 and 99 Chances in 100).

ferent, but due to the probability given, the variances were compared pairwise as were those for other properties where heterogeneity was more clearly indicated.

In order to find which variances caused the heterogeneity, the variances were paired with each other for comparison. Making use of the ratio of the squares of the variances and the transformation that the ratio is exponential $(2Z)$ and that Z has Fisher's distribution, Rider[4] gives a method of computing the probability that the two variances in question are the same. In no case was there more than one variance which caused the heterogeneity. This variance is listed as the odd variance in Table IV. When the result indicates the existence of two separate universes, that is, two degrees of scatter which cannot be due to chance, the specimens producing the odd scatter are indicated in the column headed "Odd Variance."

It is possible to calculate from Table III how many results must be averaged in order to be sure that the resulting figure falls within any desired distance from the true average of the entire lot of the material: that is, from the average of a very large number of tests. The results can be shown in the form of curves, as in Figs. 5 and 6.

It will be plain from the graphs of Figs. 5 and 6 that the number of specimens required depends upon how great a departure from the true average value is tolerable for a given purpose and also on the certainty that this error is not accidentally exceeded. The latter is expressed as a probability of chance occurrence: that is, as the odds that the error may be greater than that desired. The number of specimens required increases rapidly as the permissible error decreases or as the odds that the assumed error will be exceeded by accident decreases. It varies with individual shapes of test coupons and is not the same for each of the several static properties usually observed in the tension test.

TABLE V.—NUMBER OF TESTS REQUIRED FOR GIVEN DEGREES OF ACCURACY FOR DOUBLE KEEL BLOCK.

Probability (chances in 100)	1	5	10	20	30
Acceptable Variation	Number of Tests Required				
Yield Point					
500 psi	34	20	14	9	6
600 psi	24	14	10	6	4
700 psi	18	10	8	5	3
800 psi	14	8	6	4	3
900 psi	11	6	5	3	2
1000 psi	9	5	4	3	...
1200 psi	6	4	3	2	...
1400 psi	5	3	2	...	...
1600 psi	4	2	...	...	...
Tensile Strength					
100 psi	286	166	177	72	47
200 psi	72	42	30	18	12
300 psi	32	19	13	8	6
400 psi	18	11	8	5	3
500 psi	12	7	5	3	2
600 psi	8	5	4	2	...
700 psi	6	4	2	...	...
800 psi	5	3	...	...	...
1000 psi	3	2	...	...	...
Elongation					
1 per cent	17	10	7	5	3
2 per cent	5	3	3	2	2
3 per cent	2	2	2	...	...
Reduction of Area					
1 per cent	60	35	24	15	10
2 per cent	15	9	6	4	3
3 per cent	7	4	2	2	2
4 per cent	4	3	...	...	...
5 per cent	3	2	...	...	...
6 per cent	2	...	...	...	...

CONCLUSIONS

1. The uniformity of test results depends upon the design of the coupon that is used. There is no one single best form of coupon. Presumably the most desirable coupon design would be that having the lowest degree of scatter of the test values. However, the coupon design does not have the same order of rating for each of the four mechanical properties tested. It is, therefore, a matter of choice which property should be kept most uniform. From that choice, once made, it may be possible to select a

coupon design and base further specifications on that form alone.

2. It has been found that a surprisingly large number of tests must be made for a single heat of steel if any precise knowledge is achieved as to the true average value of the mechanical properties of that heat.

By way of illustration, Table V gives the number of specimens which must be tested and averaged to obtain a result which does not differ from the true average by an amount greater than that indicated in the column headed "Acceptable Variation." This number of specimens varies with the degree of accuracy desired and has been calculated for five different probability factors.

Thus, the first column in the table shows the number of specimens which must be tested in the event it is desired that their average will not differ from the true average (with the accepted variation) more than once in 100 trials. In like manner, the second column gives the number of specimens required if their average is not to differ from the true average (with the accepted variation) more than 5 times in 100 trials. The remaining three columns give the same data for probabilities of 10 excessive variations per 100 trials, 20 excessive variations per 100 trials, and 30 excessive variations per 100 trials.

As an example, assume that it is desired to determine how many test bars must be pulled in order to obtain average results which will not vary more than 500 psi. from the true average for tensile strength and that this result can be counted upon at least 90 times out of 100 trials. From the table, reading across from the 500 psi. in the column headed "10," it may be found that to accomplish this, 5 test bars must be pulled and their results averaged.

The actual figures differ with the type of coupon used and Table V is based on the conventional double-keel block.

Acknowledgments:

This work was conducted for and with the advice, financial support and assistance of the Technical Research Committee of the Steel Founders' Soc. of America and its Technical and Research Director, C. W. Briggs. Thanks are due that Society for permission to publish the results.

ROCKWELL C HARDNESS ON CYLINDRICAL STEEL SPECIMENS*

By Frank W. Hussey[1]

Synopsis

A series of somewhat more than 2800 individual hardness readings were made on the flat and curved surfaces of carefully prepared steel specimens of small diameter to determine the deviations of hardness readings taken on curved surfaces from the "true" values of flat areas. Eighty steel specimens of eight diameters were turned down from larger stock to assure homogeneous metal, heat treated to obtain ten ranges in hardness in each size group, and then ground to size with a fine degree of surface finish. The hardness tester was operated and calibrated in accordance with the practices recommended in A.S.T.M. Standard Methods (E 18 – 42)[2] except, that even stricter control was exercised over some variables. The resultant deviations, when plotted, indicate line relationships of such slight curvatures that straight line averages, obtained by the method of least squares, were utilized and considered sufficiently accurate for all practical purposes.

Correlation curves are presented which may be used to obtain actual Rockwell C hardness values from measurements made on cylindrical surfaces of the following diameters: $\frac{1}{4}, \frac{3}{8}, \frac{1}{2}, \frac{5}{8}, \frac{3}{4}, \frac{7}{8}$, 1, and $1\frac{1}{4}$ in.

It was observed that many specifications and drawings for round steel objects prescribed Rockwell C hardness requirements which were not met when the articles were tested on the curved surfaces. It was believed that errors of considerable magnitude were involved in measuring Rockwell C hardness on small diameter cylindrical surfaces and assuming that the values thus obtained were representative of the actual hardness of the metal. Since no usable information was found in the literature, this program was initiated to determine experimentally the deviation of the apparent from the "true" hardness when conducting tests on curved steel surfaces in the hardness range of 20 to 65 Rockwell C.

This project was chiefly suggested during a study of the results of acceptance tests on special small diameter thick-walled tubular items. The results of Rockwell C hardness tests made directly on these items were often a few points under the requirements. The accuracy of such tests in indicating the "true" hardness of the metal was questioned and a search was made for informative data on the subject. The opinions of authorities differed regarding the probable errors involved in the hardness testing of small steel cylinders when using the Rockwell C scale. Some authorities stated that there was little deviation of the apparent from the

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Frankford Arsenal, Philadelphia, Pa.

[2] Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 42), 1949 Book of A.S.T.M. Standards, Part 2, p. 1025.

"true" Rockwell C hardness for steel down to $\frac{1}{2}$-in. diameter, while others stated that the curvature should always be reported because of the error involved.

The only specific information on the subject at the initiation of this program was a group of curves which were furnished by the Wilson Mechanical Instrument Co.[3] These curves supposedly indicated the minus errors in points Rockwell C which occurred in measuring the hardness on steel cylinders from $\frac{1}{4}$ to $1\frac{1}{4}$ in. in diameter. It was requested that discretion be used in applying the curves because they were derived merely by computations after developing a formula in which many assumptions were made. The accuracy of the predicted corrections was obviously too uncertain for the direct application of the curves, but they did serve to arouse interest in the problem in view of the errors indicated. Later experimental results confirmed the theoretical calculations within reasonable limits.

The proposed investigation was also to supplement the work of W. E. Ingerson[4] who reported experimental results of hardness tests on specimens from $\frac{1}{8}$ to 1 in. diameter of materials in the hardness ranges of the Rockwell B, F, and G scales. Ingerson found that the variations in apparent hardness, as measured on flat and curved surfaces, checked the theoretical differences, in general, closer than $\frac{1}{2}$ to 1 Rockwell number.

During the course of this project the first published article on the subject appeared in *Metal Progress*.[5] This paper, by Fleischmann and Jenkins, reported the errors resulting in measuring the Rockwell C hardness of five diameters of steel cylinders from 0.250 to 0.630 in. Another contribution to the subject was a series of correction factor curves for the Rockwell B, C, 30N, and 30T scales for specimens up to one inch in diameter. These were determined emperically by David Wallace and were published in *Materials and Methods*.[6]

METHODS

Specimen Material:

A non-shrinkable deep oil-hardening steel having the following composition was used for these tests:

Carbon, per cent	0.91
Manganese, per cent	1.18
Phosphorus, per cent	0.015
Sulfur, per cent	0.012
Silicon, per cent	0.18
Nickel, per cent	0.01
Chromium, per cent	0.43
Molybdenum, per cent	0.10
Tungsten, per cent	0.48
Vanadium, per cent	0.10

Preparation of Specimens:

The homogeneity of the bar stock was determined by microscopic examinations of small sample disks. The initial decarburization was slight and extended to a radial depth of from 0.001 to 0.002 in. The examination, however, revealed zones of abnormal grain size at the surfaces of the bar stock. This zone was 0.031 and 0.046 in. deep in the $\frac{7}{8}$ and $1\frac{1}{4}$ in. diameter stock and approximately 0.015 in. deep in the other sizes. In order to provide homogeneous metal of approximately uniform grain size, the outer abnormal area was removed.

Specimens from $\frac{1}{4}$- to $\frac{7}{8}$-in. diameter were machined from stock $\frac{1}{8}$ in. larger in diameter, while 1- and $1\frac{1}{4}$-in. specimens were prepared from $1\frac{1}{4}$- and $1\frac{1}{2}$-in. diameter stock, respectively. Eighty test specimens consisting of ten of each diameter were turned down as mentioned, but left uniformally 0.005 in. oversize to allow sufficient stock for final grinding after heat treatment. This also permitted the removal of any possible decarburization which might have occurred during heat treatment.

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[3] Letter from the Wilson Mechanical Instrument Co., Inc., dated March 22, 1940, to Frankford Arsenal, attention of Mr. N. Murdza.

[4] W. E. Ingerson, "Rockwell Hardness of Cylindrical Specimens, "*Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1281 (1939). Experimental results substantiated theoretical calculations for correcting hardnesses of cylinders in the Rockwell B, F, and G scales.

[5] W. L. Fleischmann and R. S. Jenkins, "Rockwell Hardness (Diamond Penetrator) of Cylindrical Specimens." *Metal Progress*, Vol. 47, No. 2, February, 1945, pp. 275–277. Experimental results of Rockwell C hardness tests on small diameter cylindrical steel specimens, with a nomograph for finding corrections.

[6] David Wallace, "Rockwell Hardness Correction Factors," *Materials and Methods*, Vol. 23, February, 1946, pp. 471, 473 (File Facts No. 106), series of correction curves for Rockwell B, C, 30N, and 30T scales covering diameters up to 1 in.

The oversize specimens were quenched after a 20-min. soak at 1475 F. and then tempered for 2 hr. according to the following schedule:

Tempering Temperature, deg. Fahr.	Desired Hardness, Rockwell "C" Scale	Hardness Obtained, Rockwell "C" Scale[a]
200 (Stress Relief)........	64 to 65	64.7
380........................	60	61.4
600........................	55	56.3
780........................	50	50.1
870........................	45	47.1
1040.......................	40	40.6
1130.......................	35	36.3
1210.......................	30	27.6
1270.......................	25	24.1
1310.......................	20	21.3

[a] Average for the eight sizes of specimens.

The heat-treated specimens were then centerless ground to the desired diameters, and approximately the same thickness of metal was also removed from the flat surfaces on a surface grinder. The resultant diameters, within ± 0.001 in. were very close to the decimal equivalents of the fractional sizes.

Testing Procedures—Precautions Taken to Insure Accurate Results:

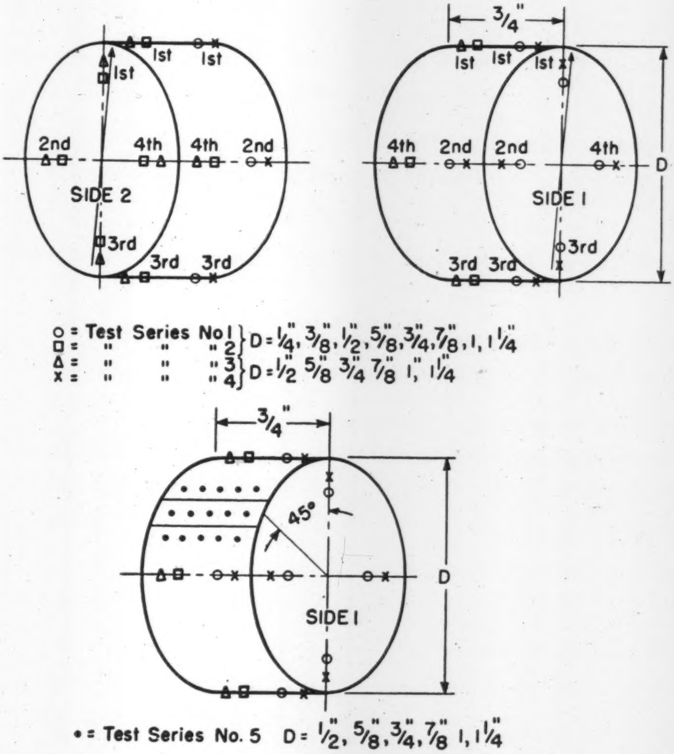
In general, the hardness determinations were made according to the procedures recommended in A.S.T.M. Standard Methods of Test for Rockwell Hardness of Metallic Materials (E 18 - 42).[2] In most instances, any departures from the recommended practices were in observing even stricter controls than are usually required. The useful range of the Rockwell C scale is from 20 upwards. Values below 20, however, were recorded and used for the curved surface hardnesses of the softest cylinders since the "true" or flat surface hardness of these specimens was approximately Rockwell C 20.

The tests were all made on a hand-operated Rockwell Hardness Tester. This machine was kept in a glass case at all times and used only for special testing. A new diamond sphero-conical penetrator was obtained and used throughout the investigation. The accuracy and surface condition of the penetrator's contour were checked before its use and occasionally during the tests by examination with a magnifying lens. The machine and penetrator were calibrated before and after each series of tests by checking the low end, high end, and the middle of the scale range against standard test blocks. The dash pot on the Rockwell Hardness Tester was checked and adjusted, if necessary, before each series of tests so that the operating handle completed its travel in from 4 to 5 sec. with no specimen in place in the machine and with an applied major load of 100 kg. In applying the minor load, A.S.T.M. Methods E 18 - 42 indicate the proper procedure if the pointer has made the correct number of revolutions and stands within ±5 divisions of the "SET" position at the top of the dial. During all of these tests, the minor load was applied so that all settings were within ±2 divisions of "SET". In order to provide the utmost uniformity in releasing the major load, an interval of 2 sec. was counted after the weight arm was completely free from the control of the dash pot before returning the operating handle to its normal position.

Rockwell hardness numbers are ordinarily sufficiently accurate when reported to the nearest scale division of the dial, which is represented by a whole number. In an effort, however, to indicate more closely the differences between hardness numbers as measured on flat and adjacent curved surfaces, the location of the pointer was estimated to tenths of dial divisions and was thus recorded and used. It was observed, during the first series of tests, that the ordinary vibration and jar caused by near-by tension or impact testing were sufficient to affect the readings, especially when estimating to tenths of dial divisions. Therefore, all subsequent tests were made at night when little or no other mechanical testing was in progress.

All except the first series of tests were conducted by the same person (the author), in order to eliminate any possible variations due to differences in the technique of operating the hardness tester.

A 2-in. diameter flat anvil was used to support the specimens for all tests on the plane surfaces. In testing the hardness on cylindrical surfaces, a suitable anvil of the V-notch type, assured correct alignment of the specimen axis with that of the penetrator. This anvil, part of the machine equipment, had two parallel horizontal steel cylinders on which the specimen was supported. Whenever an anvil was changed, the new one was seated by making a few tests on some spare material before beginning to test the specimens.


1st
1st
1st
2nd
4th
4th
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SIDE 2
3rd
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3rd
3/4"
1st
1st
1st
4th
2nd
2nd
4th
D
SIDE 1
3rd
3rd
3rd
○ = Test Series No 1
□ = " " " 2
Δ = " " " 3
x = " " " 4
D = 1/4, 3/8, 1/2, 5/8, 3/4, 7/8, 1, 1 1/4
D = 1/2 5/8 3/4 7/8 1" 1 1/4
3/4"
45°
D
SIDE 1
• = Test Series No. 5 D = 1/2, 5/8, 3/4, 7/8 1, 1 1/4

FIG. 1.—Locations of Rockwell C Hardness Tests on Flat and Adjacent Curved Surfaces of Specimens.

Precautions also were taken to provide adequate separation of indentations and to keep them at safe distances from the edges. The distance between any two impressions or the space between one of these and an edge was maintained at least equal to three times the diameter of the indentation.

Testing Procedures—Manipulations and the Treatment of Specimens During Tests:

Before beginning the hardness tests, a very light diametrical line with a small arrow at one end was scribed on one face of each specimen, as an aid in indexing the specimen and in locating adjacent flat and curved surface test positions. Five different series or sets of tests were conducted and each series on specimens of a given diameter were completed during a testing session. Figure 1 shows the general locations of testing positions on the cylindrical and flat surfaces of the blocks. In test series No. 1, as shown at the top of Fig. 1, the indentations were about $\frac{1}{4}$ in. from the edges on samples sufficiently large for this spacing. On the smaller specimens, they were closer to the edge, and greater care was required to keep the indentations a safe distance apart and away from the edge of these specimens. The first impression on the flat surface was made at the arrow end of the line but offset from it. The sample was rotated 90 deg. for each subsequent test until four determinations were made on the flat surfaces of all specimens of that size group. When the tests were completed on the flat surfaces of one or more groups, the V-notch anvil was inserted and properly seated for series of tests on cylindrical surfaces. The first test on the curved surface was made in line with the arrow or adjacent to the first test on the flat surface. The specimen was then rotated 90 deg. for the second curved surface test which was just over the side and in line with the second plane surface test, and so on until the series was completed for the entire set of specimens. The second and third test series utilized side No. 2 or the plane surface opposite to that used for the first test series. It was necessary, before proceeding with the second test series, to grind side No. 1 so as to remove the ridges surrounding the indentations and thus provide complete plane surface support for the subsequent tests. This was done in the manner of preparing metallographic specimens by working the specimens back and forth on No. 1G grade emery cloth placed on a piece of plate glass. This process was repeated on side No. 2 before the fourth test series which again employed flat side No. 1.

Upon completion of the first four series of tests, the specimens were ground in preparation for the fifth test series, to remove the ridges resulting from tests on the cylindrical surfaces. These were removed with very little change (less than 0.001 in.) in the basic diameters. A flat surface, approximately $\frac{1}{8}$ in. wide, was ground on a surface grinder in the manner shown in the lower sketch of Fig. 1. This flat, ground across the curved surface, was equidistant from previously made indentations. In the fifth set of tests, the specimen was supported in V-notch anvil for tests on both the ground flat and the adjacent curved surfaces. Great care was exercised to maintain the flat surface normal to the penetrator during the five tests made along this surface. Five hardness determinations were then made on the curved surface on both sides of the flat.

All five series of tests were conducted only on those specimens which were $\frac{1}{2}$ in. in diameter or larger. Because of their size and limited area for making the tests, the $\frac{1}{4}$- and $\frac{3}{8}$-in. specimens were carried through only the first and second series.

The differences between the higher hardness readings obtained on the flat surfaces and the somewhat lower values for the nearby curved surfaces were calculated as the testing progressed and plotted against the flat surface hardness.

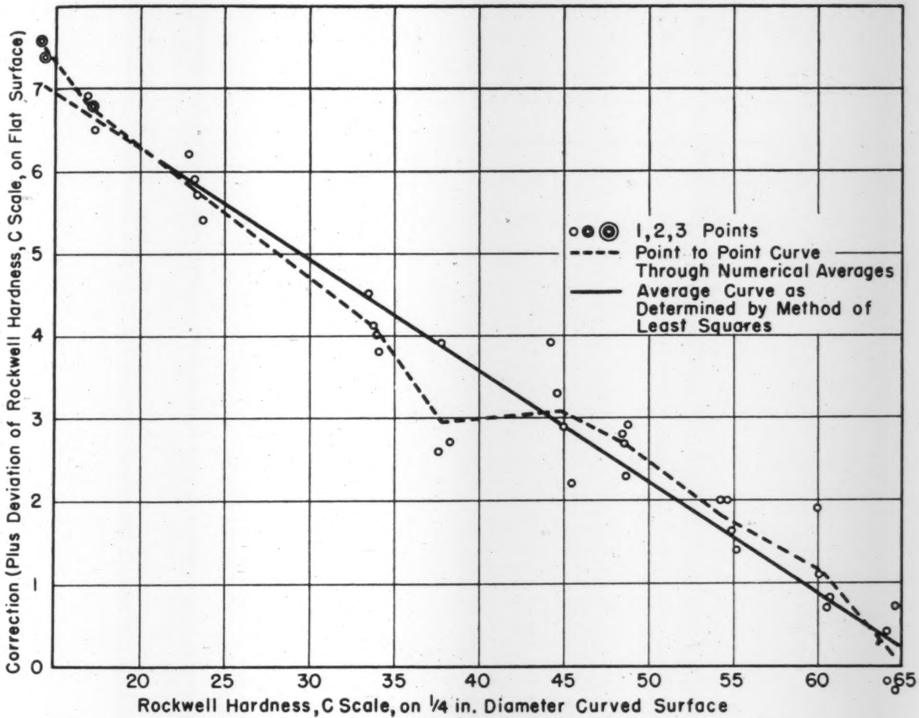

FIG. 2.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of $\frac{1}{4}$ (0.250)-in. Diameter Specimens.

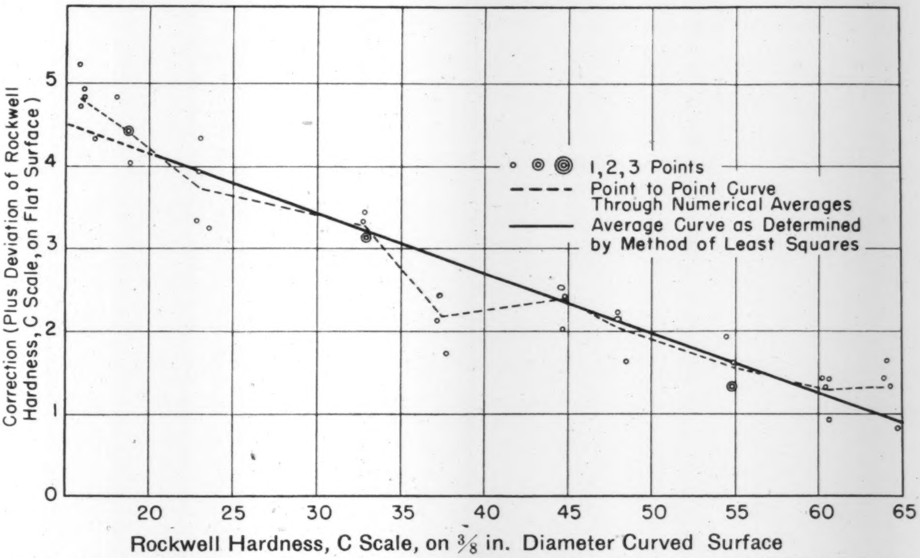

FIG. 3.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of $\frac{3}{8}$ (0.375)-in. Diameter Specimens.

As indicated in Fig. 1, two readings were made on the cylindrical surface to one on the flat in the fifth series of tests. An average of these two values obtained on the cylindrical surface was used in calculating the deviations. The results were plotted in several different ways, but the most applicable was found to be that showing the correction (plus deviation of flat surface hardness) against the hardness obtained on the curved surface. It was found necessary to use an extremely large scale to indicate with accuracy the relationship of the Rockwell C hardness on flat surfaces to the Rockwell C hardness of curved surfaces. This method of presenting the findings was therefore abandoned.

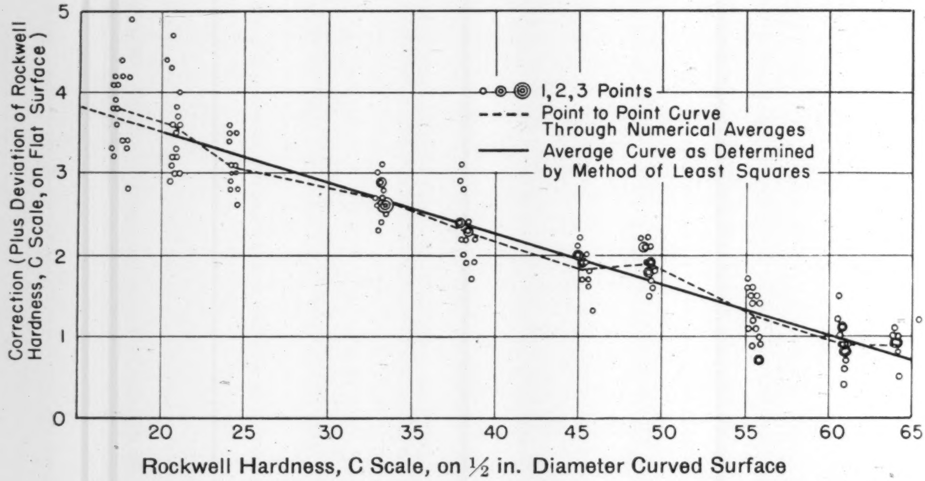

FIG. 4.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of ½ (0.500)-in. Diameter Specimens.

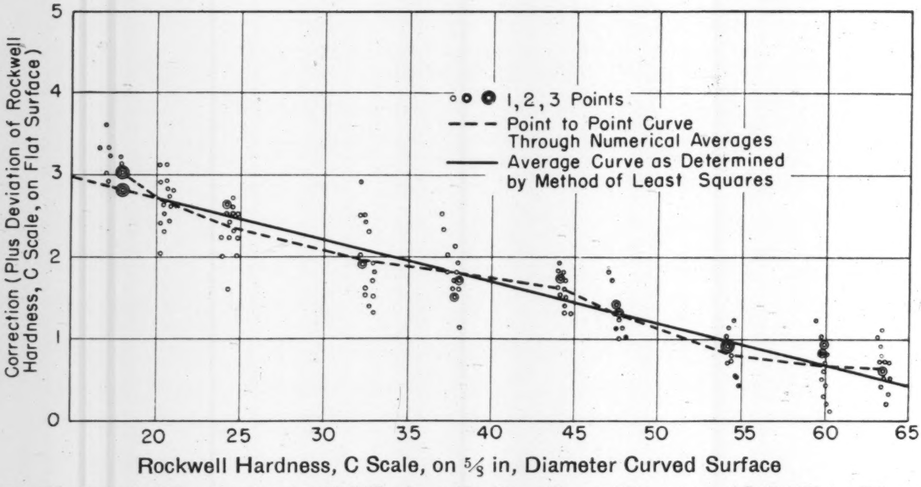

FIG. 5.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of ⅝ (0.625)-in. Diameter Specimens.

It was mentioned that inadequate control of testing conditions during the first series of tests caused discrepancies in the results of that series. In view of the uniformity of subsequent data, obtained under more carefully controlled conditions, the results of the first series were eliminated in the final analysis.

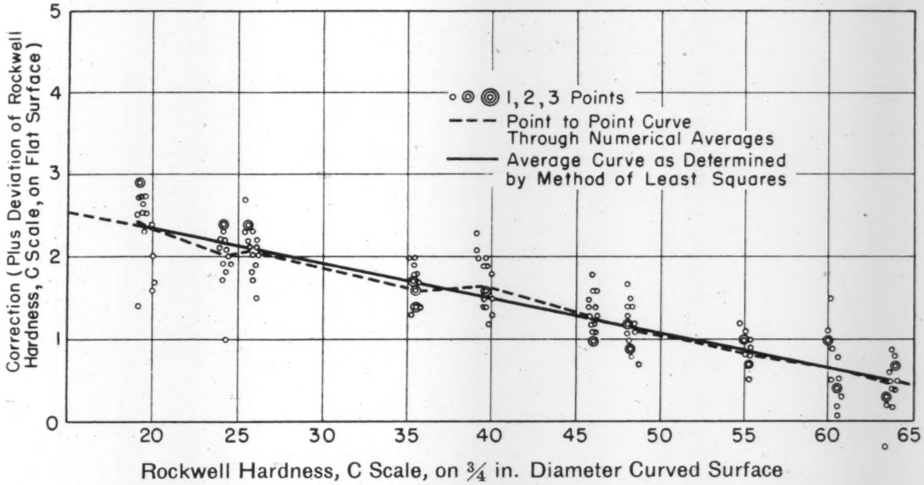

FIG. 6.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of ¾ (0.750)-in. Diameter Specimens.

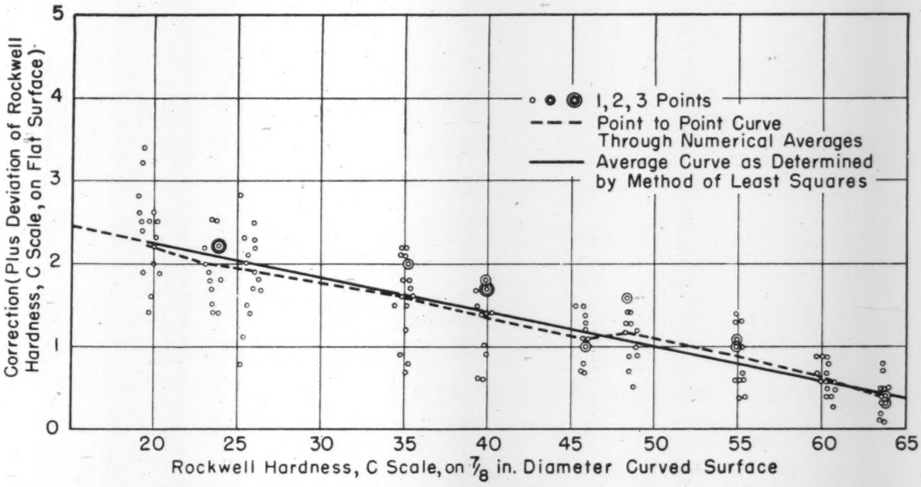

FIG. 7.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of ⅞ (0.875)-in. Diameter Specimens.

RESULTS AND CONCLUSIONS

The trends of errors indicated by the deviations of readings made on both flat and curved surfaces of the specimens are shown on Figs. 2 to 9. For practical application, "Plus corrections (Deviation of Rockwell C Hardness on Flat Surface)" are plotted as ordinates against

"Rockwell C Hardness on Cylindrical Surface" as abscissas. Each determination is indicated by a dot and one concentric circle so that the number of concentric circles signifies the number of identical values or deviations. The solid line is that obtained by the method of least squares as the one most representative of the various values. The dotted line joins the arithmetical averages of tests on individual specimens and is included only as a matter of interest. The least squares average curve for each size of specimen is replotted in Fig. 10 to provide a summary of the corrections to be added to the Rockwell C hardness readings obtained on cylinders of any of these diameters.

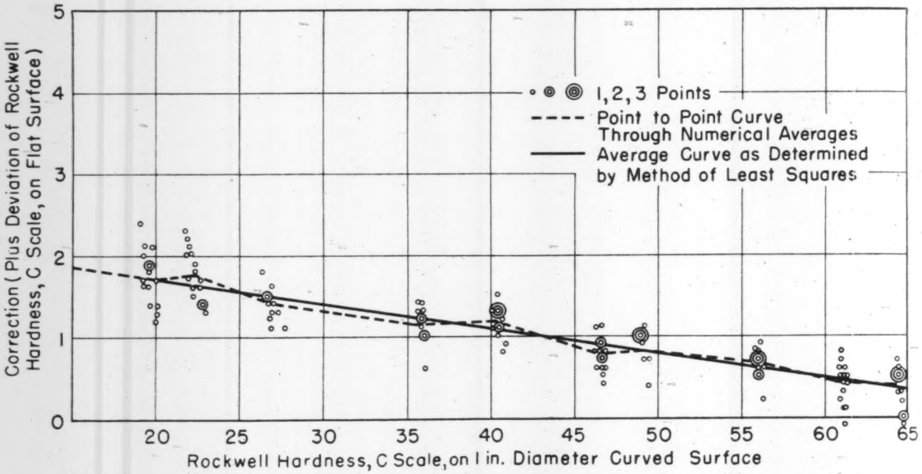

FIG. 8.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of 1 (1.000)-in. Diameter Specimens.

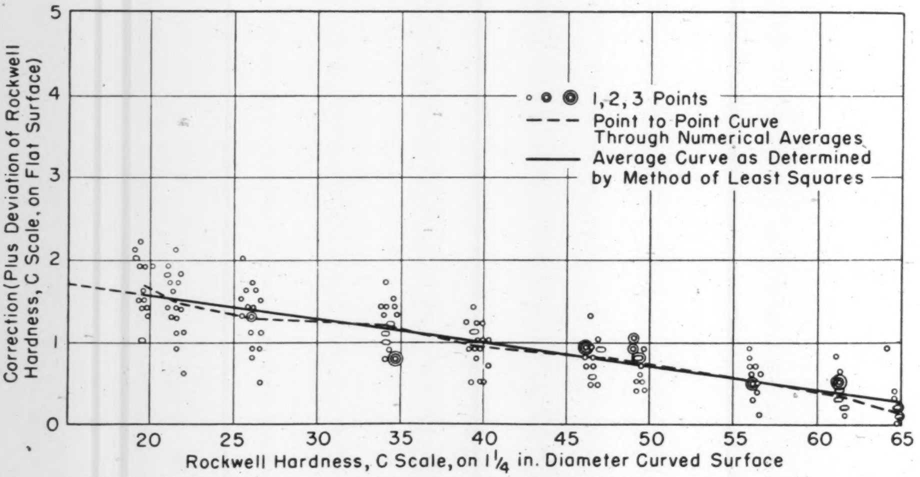

FIG. 9.—Trend of Errors in Rockwell C Hardness Readings Due to Curvature of $1\frac{1}{4}$ (1.250)-in Diameter Specimens.

The deviations of hardness values measured on curved surfaces from those obtained on flat areas are greatest at the lower end of the hardness scale and for the smaller diameters. Moreover, the curved surface hardness approaches that of the plane surface at the higher values or for the harder material. It is reasonable to assume that curves representing the true relationship would be hyperbolic such as those derived theoretically through calculations. The experimental results, however, with the normal scatter of points, indicate such slight curvatures that straight line averages are considered adequate for practical application.

It seems unreasonable for the $\frac{1}{4}$-in. curve to cross the other correction curves as it does in Fig. 10, but no satisfactory explanation for this can be made. Even though the $\frac{1}{4}$ and $\frac{3}{8}$ in. diameter curves are not representative of as many tests as are the larger sizes, these are considered adequate for practical applications.

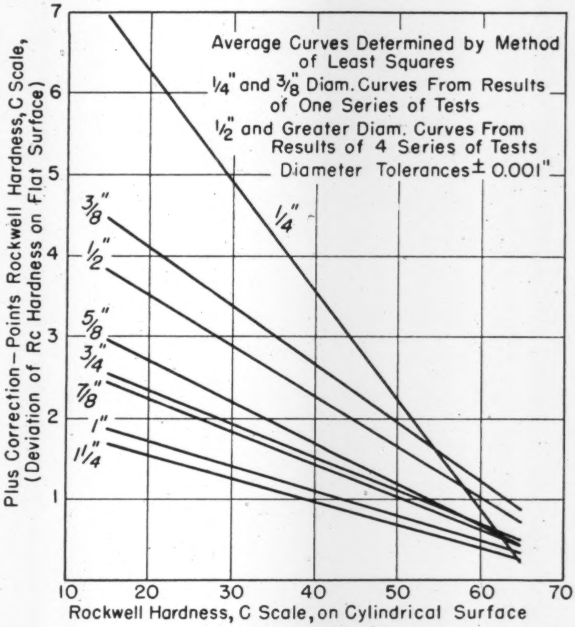

FIG. 10.—Trend of Errors in Rockwell C Hardness Due to Curvature of Various Diameters or Corrections to be Applied to Cylindrical Surface Hardness Readings.

The results obtained in this investigation exhibited practical agreement with those predicted by calculations.[3] A comparison of the two sets of curves showed the greatest divergence in the $\frac{1}{4}$ in. diameter. The average separation, however, was less than one-half a Rockwell C number, with the experimental corrections uniformly higher for the $\frac{3}{8}$ in. and larger diameters.

DISCUSSION

MR. THOMAS J. DOLAN.[1]—This is a very timely subject and the author is to be congratulated for presenting some very useful data. We have been concerned with this problem in connection with other mechanical tests (such as those presented in the paper by Sinclair and Dolan[2]) in trying to heat treat groups of specimens to the same hardness for comparative purposes. In general, it is often very convenient to be able to obtain and interpret hardness readings on the curved surfaces of cylindrical test bars.

I should like to ask a question in connection with the shape of the indentation. It occurred to me that perhaps some of the differences that have been noted in tests of different metals might possibly be related to the shape of the surface around the indentation. That is, the edge or lip surrounding the indentation in some materials may be protruded or

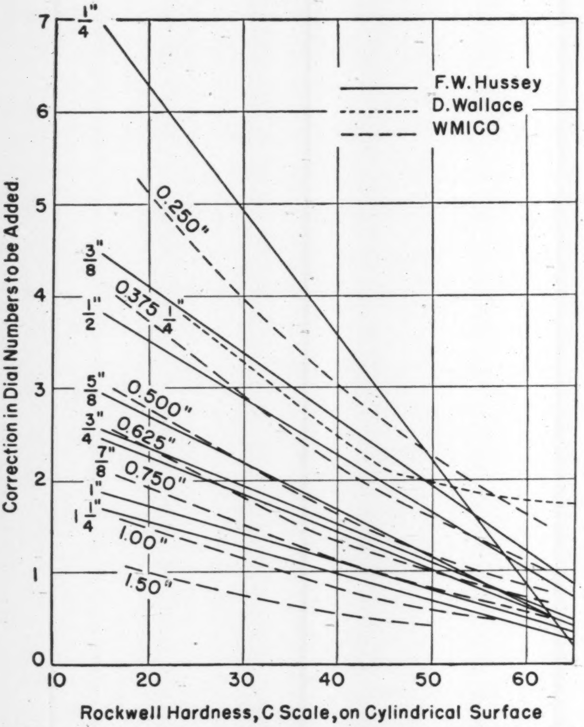

FIG. 11.—Comparison of Rockwell Hardness Results on Cylindrical Surfaces.

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[1] Dept. of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[2] G. M. Sinclair and T. J. Dolan, "Some Effects of Austenitic Grain Size and Metallurgical Structure on the Mechanical Properties of Steel," see p. 587, this publication.

raised; in other materials the indentor may cause a neighboring depression of the surface in the region of the indent.

It would seem possible that the sharpness of curvature of the test surface might influence the hardness reading to different degrees, depending upon the shape of surface contour in the region of the indentation. I should like to ask the author whether he observed any evidence of differences in type of local distortion of the perimeter of the indentation in the materials he investigated.

MR. V. E. LYSAGHT[3] (*by letter*).—This is another welcome paper to the problem of Rockwell hardness on cylindrical steel specimens. The samples were very carefully prepared and a large number of tests made.

It is very difficult to explain the crossing of the $\frac{1}{4}$-in. line. Previously published work failed to find this. Some time ago a theoretical study of this problem was made by the Wilson Mechanical Instrument Co. The curves are shown plotted in Fig. 11 superimposed over Hussey's work. It will be noted that there is generally good agreement except for the $\frac{1}{4}$-in. diameter.

Wallace[4] made a very complete study of this. To add his curves to the chart would mean the curves would all run together at the higher values. However, his $\frac{1}{4}$-in. curve is shown and it seems to indicate an opposite tendency.

MR. F. W. HUSSEY (*author's closure*).—In reply to Mr. Dolan, the investigation was concerned with only one type of material. The 80 specimens were all made of the same non-shrinkable oil-hardening steel. There were no significant differences observed in the type of indentation. All impressions were of the raised rim or "ridging" type.

Mr. Lysaght had good reason to question the results derived for the $\frac{1}{4}$-in. diameter. This inconsistency has also plagued the author, who conducted the tests, but observed nothing irregular in taking hardness readings on the $\frac{1}{4}$-in. diameter specimens. The possibility of instability, which had been suggested, was ruled out in view of the careful specimen preparation, and precisely square ends. In calculating the least-square average curves, the elimination of values below Rockwell C 20 caused only a very slight change in the slope of the curve. In view of the relatively good agreement in all other sizes, the author recommends caution in the use of his data for the $\frac{1}{4}$-in. diameter, and additional tests on this size of specimen.

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[3] Ass't. to the President, Wilson Mechanical Instrument Co., Inc., New York, N. Y.

[4] David Wallace, "Indentation Hardness Testing," Reinhold Publishing Co., New York, N. Y.

A METHOD FOR MAKING TENSION TESTS OF METALS USING A MINIATURE SPECIMEN*

By R. L. Templin[1] and W. C. Aber[1]

Although the A.S.T.M. Standard Methods of Tension Testing of Metallic Materials (E 8 - 46)[2] do not specifically impose a minimum limit on the size of specimen that may be used, the smallest one illustrated is 0.252 in. in diameter and about 3 in. long. Federal Specifications QQ-M-151a recognize a specimen as small as 0.113 in. in diameter with an over-all length of 1⅝ in. Occasionally it is necessary to determine the tensile properties of metal products when only small samples are available or when the dimensions of the pieces in one or two directions are such as to preclude the use of tension test specimens of the usual sizes.

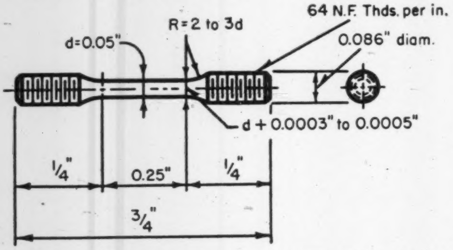

Fig. 1.—Miniature Size Tension Test Specimen, 0.05-in. Diameter.

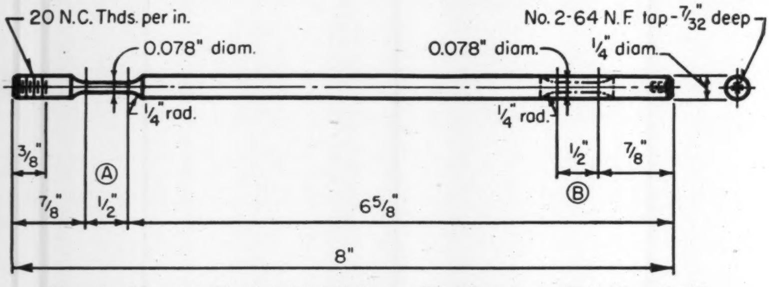

Fig. 2.—Tension Shackle for Miniature Test Specimen (Two Required).

In developing and using small tension test specimens, it must be recognized that results obtained from tests of metals having very coarse grains, that is, grains that are large in relation to the cross-section of the specimen, in all probability will not check closely the results obtained from tests of the same material when using large size specimens. If, however, both small and large specimens are used for testing material having relatively fine grains and the specimens are geometrically similar, results from the different sizes of specimens should be in reasonable agreement, provided the material is homogeneous or that the specimens are similarly located and oriented with respect to the sample from which they are taken, and similar technique is used in testing the specimens.

The Test Specimen and Equipment

The small tension test specimen studied in this investigation (Fig. 1) has a reduced section 0.05 in. in diameter by $\frac{1}{4}$ in. long and has an over-all length of $\frac{3}{4}$ in. The threaded ends are 0.086 in. in diameter with 64 threads per inch. Elongation is measured over a gage length of 4*D* (0.2 in.). The specimen is proportional to the standard $\frac{1}{2}$-in. diameter specimen having a 2-in. gage length and an over-all length of $5\frac{1}{2}$ in.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Aluminum Research Laboratories, Aluminum Company of America, New Kensington, Pa.

[2] 1949 Book of A.S.T.M. Standards, Part 2, p. 993.

A specimen of the size just indicated requires that considerable attention be accorded the design of the tension shackles to be used, in order to realize the required uniform and axial loading of the specimen. The tension shackles developed for this purpose are shown in Fig. 2. The reduced section at *A* serves to function instead of the more commonly used spherical joint in such shackles. The use of an additional similar reduced section as shown by the dotted lines at *B* might further improve the test results obtained, particularly if the female threads in the end of the shackles were not closely coincident with the longitudinal axes of the shackles. The elastic strength of the reduced portions of the shackles, of course, should be greater than the breaking strength of the specimens to be tested. The shackles are attached to the spherically-seated fixtures used for testing the standard specimens, by means of threaded bushings.

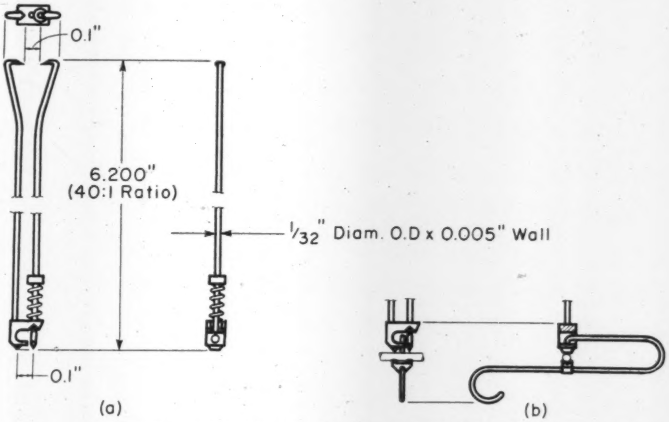

FIG. 3.—0.1-in. Strain Gage and Attaching Clamp.

Tensile strength and elongation values, using the small specimen, are relatively easy to determine, although it is necessary to be very careful in measuring the diameter of the reduced section, applying gage marks and measuring the distance between them both before and after fracture of the specimen.

In order to determine yield strength, using such a small specimen, a very small strain gage is necessary. The strain gage developed for this purpose is shown in Fig. 3(*a*). It has a 0.1-in. gage length and a mechanical magnification of approximately 40× and is similar in design to one described by Peterson and Wahl,[3] but is of simpler construction in that it is comprised of only seven parts. The gage is held on the specimen by means of the clamp shown in Fig. 3 (*b*). The total weight of gage and clamp is 0.03 oz. The extension occurring in the specimen during testing is magnified by the strain gage, then measured with a Zeiss shop type measuring microscope which reads directly to 0.01 mm. and by estimation to 0.002 mm. The complete arrangement of specimen, shackles, strain gage and microscope, ready for making a tension test, is shown in Fig. 4.

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[3] R. E. Peterson and A. M. Wahl, "Two- and Three-Dimensional Cases of Stress Concentration, and Comparison with Fatigue Tests," *Journal of Applied Mechanics*, Vol. 3, No. 1, March, 1936, pp. A-15 to A-22.

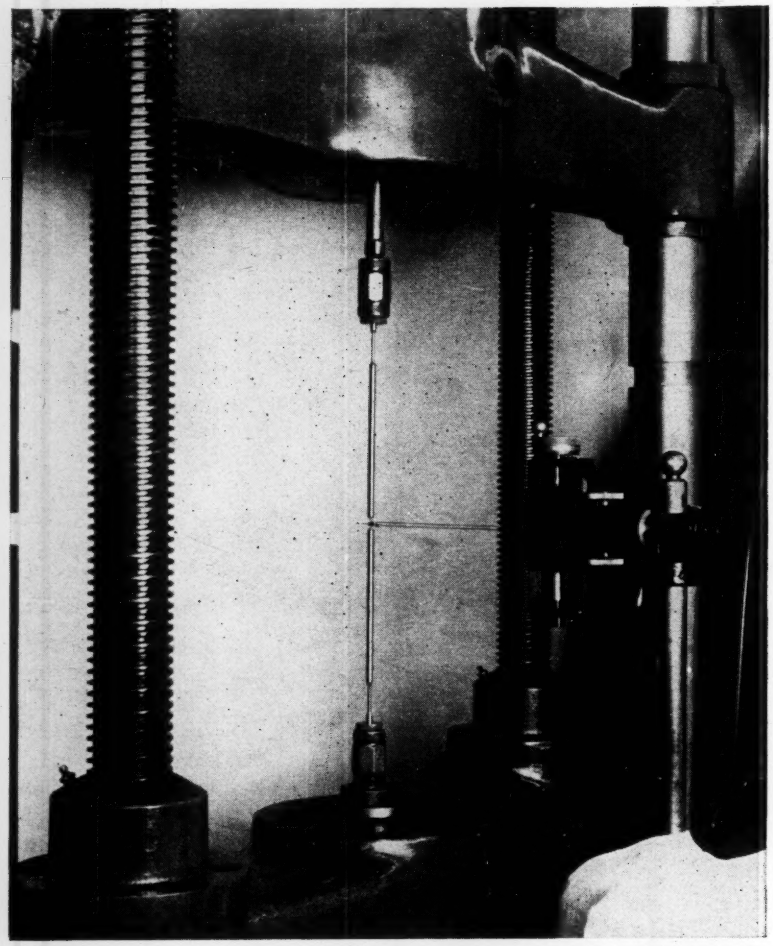

FIG. 4.—Arrangement of Apparatus for Tension Stress-Strain Test of Miniature Specimen (0.05 in. Diameter).

TEST PROCEDURE

The procedure for making tension tests with the small specimen is similar to that used for the larger specimens, but extreme care must be exercised throughout the tests because of the delicacy of the specimen and some of the equipment used.

The following method of applying gage marks to an 0.05-in. diameter test specimen differs from that used for specimens of larger diameter but was necessary to prevent damage to the small specimen. The specimen was machined to size and removed from the lathe, after which the diameter of its reduced section was measured by means of Brown & Sharpe jewelers' micrometer calipers which could be read directly to 0.0001 in. After the diameter was measured, the reduced section was coated with lay-out fluid and the specimen was returned to the fixture for holding it in the chuck of the lathe. Gage marks were placed upon the reduced section 0.2 in. apart by marking through the lay-out fluid by means of a sharp orangewood stylus held in the tool post of the lathe. The stylus was moved from the position for placing one gage mark to the position for placing the other gage mark by means of the micrometer screw of the compound rest of the lathe. Both before and after fracture, the distance between the gage marks on the specimen is measured by means of the measuring microscope previously mentioned.

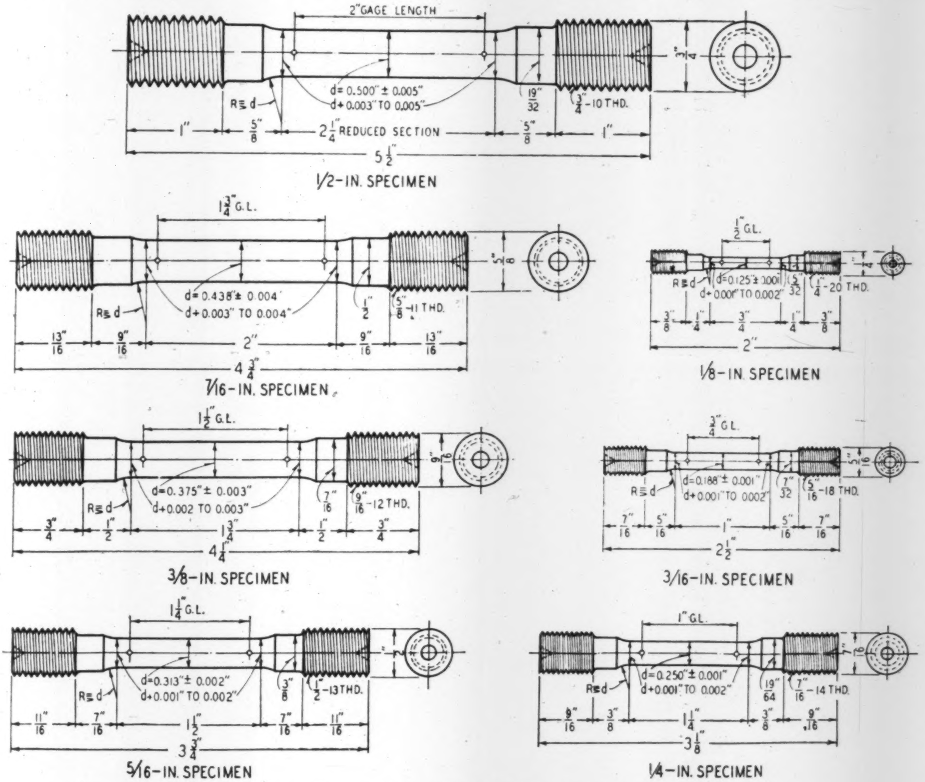
2" GAGE LENGTH
d= 0.500" ± 0.005"
d+0.003" TO 0.005"
R≡d
19/32
3/4-10 THD.
1"
5/8
2 1/4" REDUCED SECTION
5/8
1"
5 1/2"
3/4
1/2-IN. SPECIMEN
1 3/4" G.L.
d= 0.438"± 0.004"
d+0.003" TO 0.004"
R≡d
1/2
5/8-11 THD.
13/16
9/16
2"
9/16
13/16
4 3/4"
5/8
7/16-IN. SPECIMEN
1/2" G.L.
R≡d
d=0.125"± 0.001"
d+0.001" TO 0.002"
5/32
1/4-20 THD.
3/8
1/4
3/4
1/4
3/8
2"
1/8-IN. SPECIMEN
1 1/2" G.L.
d=0.375"± 0.003"
d+0.002 TO 0.003"
R≡d
7/16
9/16-12 THD.
3/4
1/2
1 3/4
1/2
3/4
4 1/4"
9/16
3/8-IN. SPECIMEN
3/4" G.L.
d=0.188"± 0.001"
d+0.001" TO 0.002"
R≡d
7/32
5/16-18 THD.
7/16
5/16
1"
5/16
7/16
2 1/2"
5/16
3/16-IN. SPECIMEN
1 1/4" G.L.
d=0.313"± 0.002"
d+0.001" TO 0.002"
R≡d
3/8
1/2-13 THD.
11/16
7/16
1 1/2"
7/16
11/16
3 3/4"
5/16-IN. SPECIMEN
1" G.L.
d=0.250"± 0.001"
d+0.001" TO 0.002"
R≡d
19/64
7/16-14 THD.
9/16
3/8
1 1/4"
3/8
9/16
3 1/8"
7/16
1/4-IN. SPECIMEN

FIG. 5.—Proportional Sizes of Round Tension Test Specimens.

Note.—In general, the largest possible size of specimen should be used. Reduced section has gradual taper from ends to middle.

Because of the small size of the specimen and the delicacy of the strain gage, it is very difficult to mount the strain gage on the specimen at an exact gage length of 0.1 in. However, for the determination of yield strength of a material whose modulus of elasticity is known, it is not necessary to set the strain gage precisely to a given gage length nor to know the exact magnification of the strain gage. It is sufficient to mount the strain gage at approximately the correct gage length and then to apply increments of load and record the corresponding strain-gage readings. After the data are obtained, the load and strain-gage readings are plotted and the load corresponding to the yield strength may be determined from the resulting curve when the modulus of elasticity of the material and the original area of the specimen are known.

Although the exact magnification of the strain gage need not be known for determination of yield strength by this method, the strain gage was calibrated previously and was found to indicate readings within satisfactory limits of accuracy.

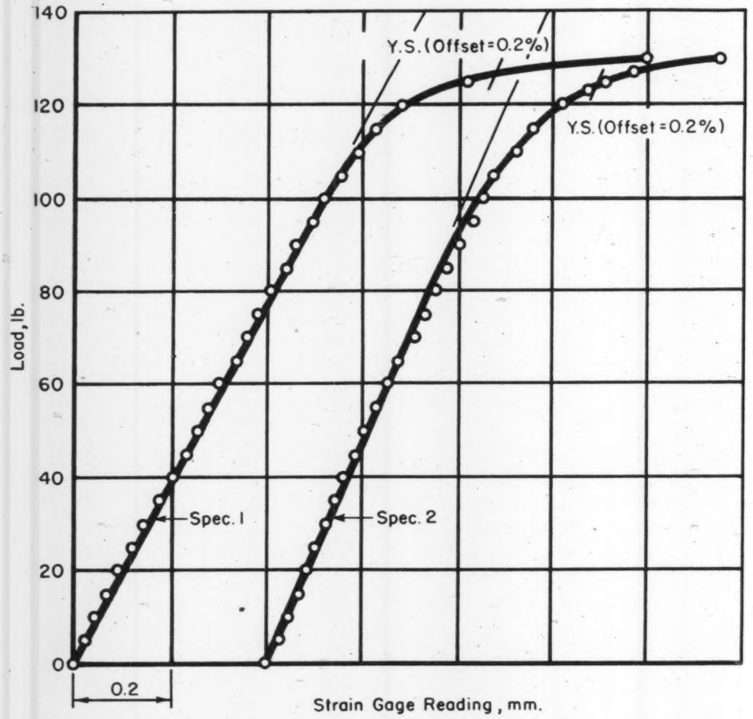

FIG. 6.—Tension Load-Strain Curves of Aluminum Alloy 14S-T6 Rod Obtained from Tests Using Miniature Specimens.

In order to compare the results obtained from the miniature specimen with similar data from larger specimens, various sizes of the tension specimens ranging in diameter from ½ in. to ⅛ in. (Fig. 5) were machined from the same sample of aluminum alloy 14S-T6 rolled rod. The axes of all the specimens were parallel to the direction of rolling of the rod and were located midway between the center and the outside surface of the rod. Two complete sets of eight specimens each were prepared to furnish duplicate results for each size of specimen.

The larger specimens were tested using an Amsler type 10 SZBDA machine with seven different capacities ranging from 200 lb. to 20,000 lb. Previous calibration in accordance with A.S.T.M. Standard Methods[4] showed this machine to give correct loads within 1 per cent.

The miniature specimens were tested in Southwark-Tate-Emery Universal Testing Machine No. 50-T-E-162 which was provided with three capacity ranges of 500, 5000, and 50,000 lb. Only the smallest load range was used. From previous calibration of this machine it was known to give load readings for this range within less than 0.4 per cent. The setup for testing the miniature tension specimen is shown in Fig. 4. The support for the measuring microscope rested on the lower platen of the testing machine in such a position that the microscope could be focused upon both arms of the strain gage simultaneously. The testing procedure consisted of obtaining readings by means of the microscope for each arm of the strain gage with no load on the specimen and then additional readings for each increment of load applied. Increments of load were applied and corresponding strain readings observed and recorded until the applied load exceeded the yield strength of the material. The strain gage was then removed from the specimen and loading continued to failure. It may be pointed out that in the tests using the miniature tension specimen two operators are required when making the strain measurements. About 9 man hours are required for preparing the specimen, testing, recording, and reducing the data for the miniature tension specimen as compared to about ⅔ man hour required for making a similar test using the standard ½-in. diameter threaded-end specimen and an autographic extensometer.

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[4] Tentative Methods of Verification of Testing Machines (E 4-47 T), 1949 Book of A.S.T.M. Standards, Part 1, p. 1223.

TABLE I.—MECHANICAL PROPERTIES OF 14S-T6 ALUMINUM ALLOY ROLLED ROD, DETERMINED USING DIFFERENT SIZES OF TENSION TEST SPECIMENS.

Nominal Specimen Size, in.	Specimen	Diameter, in.	Area, sq. in.	Tensile Strength		Yield Strength, 0.2 per cent offset		Elongation in 4*D*		Reduction of Area, per cent
				psi.	[a]	psi.	[a]	per cent	[a]	
½	No. 1	0.500	0.1963	72 100	+1400	65 000	+1300	11.5	+0.3	27
	No. 2	0.500	0.1963	71 300	+600	65 000	+1300	11.0	−0.2	26
				71 700	+1000	65 000	+1300	11.2	0	26
7/16	No. 1	0.434	0.1479	71 000	+300	64 200	+500	10.9	−0.3	26
	No. 2	0.434	0.1479	70 000	−700	62 500	−1200	10.9	−0.3	26
				70 500	−200	63 400	−300	10.9	−0.3	26
3/8	No. 1	0.373	0.1093	70 300	−400	62 900	−800	11.3	+0.1	26
	No. 2	0.374	0.1099	71 900	+1200	64 600	+900	11.3	+0.1	25
				71 100	+400	63 800	+100	11.3	+0.1	26
5/16	No. 1	0.311	0.0760	70 900	+200	63 800	+100	10.4	−0.8	27
	No. 2	0.312	0.0765	70 700	0	63 700	0	11.2	0	25
				70 800	+100	63 800	+100	10.8	−0.4	26
¼	No. 1	0.2505	0.0493	71 800	+1100	64 900	+1200	11.0	−0.2	28
	No. 2	0.2505	0.0493	70 800	+100	64 400	+700	11.0	−0.2	26
				71 300	+600	64 600	+900	11.0	−0.2	27
3/16	No. 1	0.1865	0.0273	69 400	−1300	62 600	−1100	10.7	−0.5	26
	No. 2	0.1865	0.0273	70 300	−400	63 700	0	10.7	−0.5	26
				69 800	−900	63 200	−500	10.7	−0.5	26
⅛	No. 1	0.1257	0.0124	69 500	−1200	62 100	−1600	12.0	+0.8	29
	No. 2	0.1254	0.0124	69 700	−1000	62 100	−1600	12.0	+0.8	28
				69 600	−1100	62 100	−1600	12.0	+0.8	28
0.050	No. 1	0.0500	0.00196	71 200	+500	64 600	+900	12.7	+1.5	32
	No. 2	0.0500	0.00196	69 600	−1100	63 400	−300	11.1	−0.1	35
				70 400	−300	64 000	+300	11.9	+0.7	34
Average of all specimens				70 700	....	63 700	....	11.2	....	27

[a] Deviation from average of all specimens.

RESULTS OBTAINED

The load-deformation data obtained from the tension tests using the miniature specimen are shown in Fig. 6. The load scale can be converted to stress by dividing by the cross-sectional area of the specimen. The slope of the initial portion of each curve, expressed in pounds per square inch, is the tensile modulus of elasticity of the material, 10,500,000 psi.[5] The strain scale, in inches per inch, can then be evaluated from the stress and the modulus. The 0.2 per cent offset is then readily located as shown on the curves, and the yield strengths obtained in accordance with Standard Methods of Tension Testing of Metallic Specimens (E 8 – 46).[2] The actual slopes of the curves for specimens 1 and 2 are different, although the modulus of elasticity values are the same, because the initial gage length to which the strain gage was set was slightly different for each specimen.

The larger diameter tension specimens from the same material were carefully prepared, measured, and tested in accordance with the A.S.T.M. Standard Methods and the yield strength values were determined from autographic records made by means of a Templin autographic extensometer.[6]

The mechanical property values obtained from the tension tests of the specimens are given in Table I. The data show that the values from the tests of the 0.05-in. diameter specimens are in very good agreement with the average values for all of the specimens tested. The largest difference found occurred in the reduction of area values where it may be observed that those from the 0.05-in. diameter specimens are greater than the average values for all the specimens. In general, the values for the different sizes of test specimens are in good agreement. In no case did the tensile strength value for any specimen of the entire group deviate from the average for all of the specimens by more than 2.0 per cent. The maximum deviation of any of the yield strength values from the average for all specimens was only slightly greater, 2.5 per cent. The elongation values for the different specimens ranged from 10.7 to 12.6 per cent in 4*D*. The reduction of area values are approximately the same, within close limits, for specimens from ½ in. to 3⁄16 in. in diameter but are slightly greater for the ⅛-in. and 0.05-in. diameter specimens.

From the results just discussed it has been concluded that:

1. Miniature size tension test specimens 0.05 in. in diameter can be used satisfactorily for the determination of tensile strength, yield strength, elongation in 4*D*, and perhaps reduction of area of at least some metals provided the material has a relatively fine grain structure and provided the modulus of elasticity of the material has been established.

2. The errors in the mechanical properties obtained using the miniature size specimen need not be greater than those encountered when using the standard ½-in. diameter tension specimen provided proper attention is accorded the details of the testing procedure used.

3. The cost of making tension tests using the miniature size specimen may be appreciably greater than the cost when using the standard ½-in. diameter specimen, particularly when determining yield strength values; but the cost of determining only tensile strength and elongation in 4*D* using the miniature specimen is probably not appreciably greater than when using the standard ½-in. diameter specimen.

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[5] R. L. Templin and E. C. Hartmann, "The Elastic Constants for Wrought Aluminum Alloys," *Technical Note No. 966*, Nat. Adv. Committee Aeronautics, January, 1945.

[6] R. L. Templin, "An Automatic Autographic Extensometer for Use in Tension Tests of Materials," *Proceedings*, Am. Soc. Testing Mats., Vol. 32, Part II, p. 783 (1932).

DISCUSSION

MR. WALTER RAMBERG[1] (*presented in written form*).—Messrs. Templin and Aber have shown us that it is possible to scale down the standard round tension specimen to one tenth its linear dimensions and obtain values of yield strength, tensile strength, and elongation on the miniature specimen that show no more scatter than for the standard specimen. This reflects a care in machining and in testing technique that is not found in many places outside the Aluminum Research Laboratories.

I should like to ask the authors if special difficulties were encountered in machining the specimens and also if unusual precautions had to be taken during the test to prevent errors in strain readings because of building vibration. How is damage to the tension shackles prevented because of the rebound of the specimen after rupture in the case of materials of low ductility?

MR. W. C. ABER (*author*).—The small specimens are prepared in our shop by skilled instrument makers and no special difficulties are encountered in their preparation. Because of the delicacy of the specimens, extreme care must be used in the machining operations.

We are not bothered by building vibration probably because our testing machines are located on the ground floor. We do take the precaution of having no tests conducted in other machines in the same room while making tension tests of miniature specimens.

The shackles have not been damaged by rebound of the specimens at the time of rupture because the loads involved have been relatively small and all of the specimens tested have had at least 2 per cent elongation.

MR. S. A. GORDON.[2]— I am curious as to just what kind of results you would get if you ran these tests in crossgrain, or a very fine grain structure. What effect would the size have on that?

MR. ABER.—Provided the grain structure is fine, the effect of size of test specimen should be the same regardless of whether all of the specimens have been prepared across grain or all with grain. Differences in results may be expected, however, between specimens cut across grain and specimens cut with grain from the same material if the material has been worked mainly in one direction. The following tabulation of results of tension tests of a $\frac{3}{4}$-in. diameter 75S-T6 aluminum alloy rolled rod shows the agreement between test results obtained from two different sizes of specimens cut with grain, or parallel to the direction of rolling, and the differences in results obtained between specimens of the same size cut with grain and across grain from the same rod.

ALUMINUM ALLOY 75S-T6 ROLLED ROD, ¾-IN. DIAMETER

Diameter of Specimen, in.	Tensile Strength, psi.	Elongation in 4D, per cent	Direction of Specimen
½	81 300	15.0	With grain
0.05	80 500	12.2	With grain
0.05	75 400	5.5	Across grain

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[1] Chief of Mechanics Div., National Bureau of Standards, Washington, D. C.

[2] Chief Structural Test Engineer, The Glenn L. Martin Co., Baltimore, Md.

THE DEVELOPMENT OF A FLAW DETECTOR FOR TUBES[1,2]

BY R. D. KODIS[3]

SYNOPSIS

The alternating current bridge method of magnetic flaw detection was evaluated by application to smooth bore tubes. Practical difficulties with the alternating current bridge method led to the investigation of the induction method, and by the proper combination of detector head design and a recorder capable of displaying three variables, cracks are shown in picture form for easy evaluation. This laboratory equipment has demonstrated that the induction method has greater speed, reliability, definition, and sensitivity than present methods for inspecting gun tubes, and can be engineered into a production inspection tool.

This paper is a progress report covering the development of a method and instrumentation for the detection of cracks in smooth bore tubes. A prior report by C. H. Hastings[4] covering the first part of this project will not be reviewed, except as to the principle used. The use in the shop, as described herein, of the flaw detector reported by Hastings,[4] led to the continuation of the project in an effort to overcome the obstacles encountered in production application of the method.

In Hastings' development and in the re-evaluation of the problem after shop use of the instrumentation, many physical phenomena were surveyed as a basis for development of a suitable flaw detector. Among the feasible techniques that were investigated, the most promising depended upon the evaluation of magnetic leakage fields due to the flaws when the tube was magnetized, and the principles employing search coil detectors were deemed to be most easily developed into the desired flaw detector. A brief description of the test procedure follows.

The tube is magnetized by passing a direct current through a conductor threaded through the tube bore. This cable is removed and the search unit employed to examine the residual circular magnetic field. A longitudinal field can be obtained in the tube by the well-known technique of placing a current-carrying coil around the circumference of the tube.

EVALUATION OF THE ALTERNATING CURRENT BRIDGE METHOD

One of the principles that has been evaluated in the development of this flaw detector is based upon the action of a ferrous cored coil having an a-c. signal on its winding when placed in a magnetic field. The magnetic field changes the magnetic characteristics of the core material, and thus the in-

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[1] Presented at a meeting of Committee E-7 on Non-Destructive Testing, held at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[2] The statements and opinions expressed in this paper are those of the author, and do not necessarily represent the views of the Ordnance Department.

[3] Physicist, Watertown Arsenal, Watertown, Mass.

[4] C. H. Hastings, "A New Type of Magnetic Flaw Detector," *Proceedings*, Am. Soc. Testing Mats., Vol. 47, pp. 651–664 (1947).

ductance of the coil. If the coil is part of a bridge network, which has been balanced at zero magnetic field or any arbitrary reference level, a change in magnetic field intensity is detectable as a change of inductance of the coil, as measured by the amount of unbalance in the bridge network. This method will be referred to as the a-c. bridge method and a block diagram of this instrument is shown in Fig. 1.

The development of an instrumentation based on the a.-c. bridge method has been reported.[4] A hand-manipulated model of this flaw detector was used for production inspection of tubes in comparison with an optical boroscope. The correlation between the two methods with respect to cracks detected was high, but it should be noted that only a small number of cracks were observed in the total number of tubes examined. The operator of the flaw detector found it difficult to distinguish crack signals from signals believed to have been produced by non-rejectable conditions. A study of a forged tube indicated five different areas where flaw-like signals prevailed. Inspection of the tube by use of an optical boroscope revealed no flaws open to the surface. Cutting and studying certain sections by macro- and micro-etching the cross-section revealed no inhomogeneities. After heat treatment of the remaining sections of the tube, the "flaws" could not be detected magnetically. Since the previous history of this tube included a cold straightening operation, it was concluded that the inhomogeneities were stress concentrations.

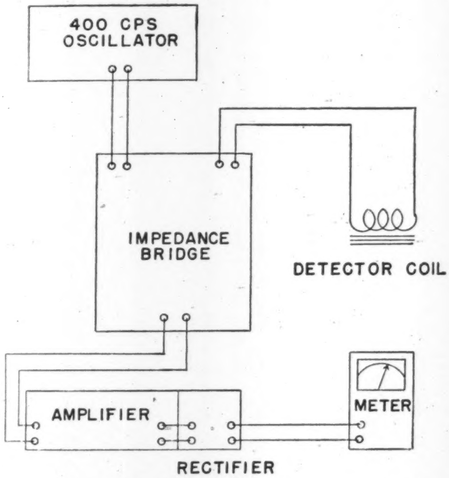

FIG. 1.—Block Diagram of A-C. Bridge Method Instrument.

In addition to the difficulty in evaluating the data, objections to the electrical instability of the instrument and its sensitivity to mechanical vibrations were offered by the operator.

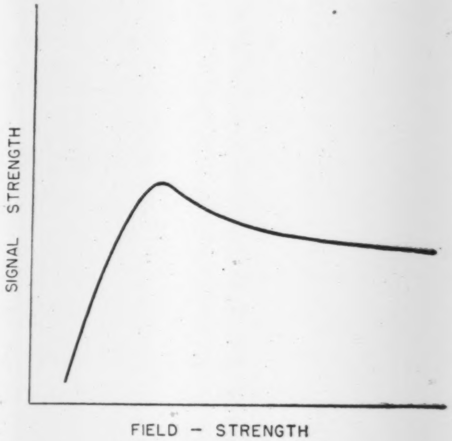

FIG. 2.—Response of A-C. Bridge Method.

The a-c. bridge method had the following inherent difficulties:

1. The poor definition or broad signals that made it difficult to locate the exact position and number of cracks.

2. The similar appearance of crack signals, and signals from other types of inhomogeneities.

3. The instability of the balance point, making the reproducibility of data difficult.

4. A limited range of magnetic field intensities over which the detector

could operate, before the relationship of field intensity and signal magnitude became an inverse instead of direct relationship (see Fig. 2).

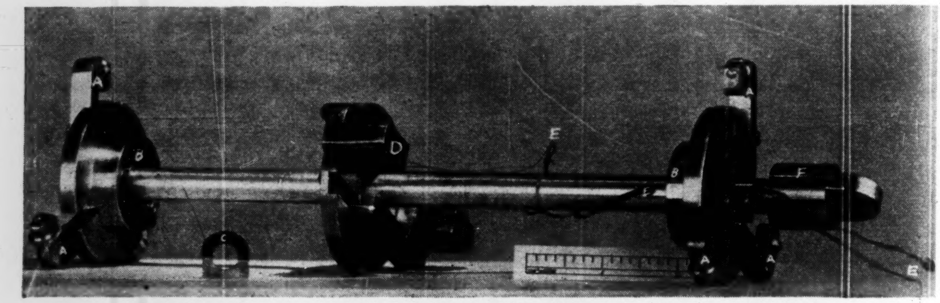

FIG. 3.—Equipment for Supporting and Revolving the Detector in Tubes.

A–Supports
B–Bearing
C-Closed Core Detector
D–Coil Holder
E–Coil Leads
F–Drive Shaft Coupling

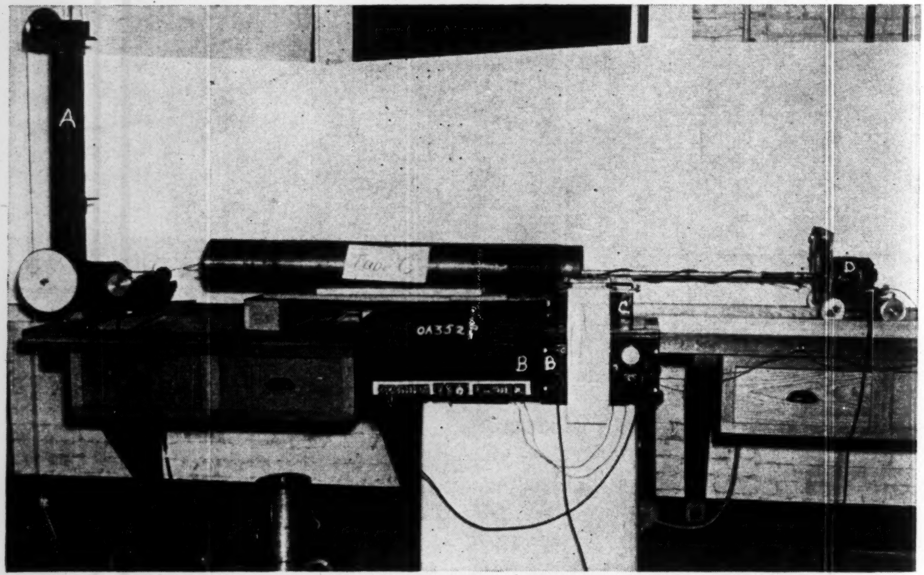

FIG. 4.—Equipment for Rotating the Detector and Moving it Longitudinally in Tube.

A—Device to Provide Longitudinal Movement *B*—Amplifiers *C*—Recorder *D*—Rotating Mechanism, Motor, and Slip Rings.

Several arrangements of detectors and coils were tried in order to overcome the above obstacles. One successful scheme to handle the overloading effect shown in Fig. 2 consisted of using a d-c. current through a coil around the detector, so as to counteract the field due to the flaw, and thus maintain the bridge in balance. The current in the bucking coil was used as a measure of the magnitude of the leakage field. Although this scheme prevented the occurrence of the overloading effect, the other difficulties enumerated which made the flaw detector unsatisfactory as a shop tool seemed to be inherent with the a-c. bridge method of flaw detection. The induction method was turned to as a possible solution for a practical flaw detector.

INVESTIGATION OF INDUCTION METHOD

The induction method, first applied to non-destructive inspection by R. L. Sanford and W. B. Kouwenhoven[5] depends upon relative motion between the search coil detector and the magnetic leakage field due to the flaw. The signals induced in the search coil can be related to the flaw causing the magnetic leakage field.

Equipment was constructed for holding a search coil and scanning it over the bore surface of the tube. Figure 3 shows the device for supporting the detector coil while it is revolved about the axis of the tube, close to the bore surface. The equipment shown in Fig. 4 was used for rotating the drive shaft and at the same time advancing the detector longitudinally down the tube. A detector with closed core, shown in Fig. 5, was mounted and its performance studied in tube *C*. Tube *C* is the thick-walled tube mentioned in the prior report,[4] and has been used as a standard for comparing detectors and checking the operation of equipment. The signals seen on a cathode-ray oscilloscope correlated well with the number of cracks visible to the unaided eye in tube *C*. Figure 6 shows wave-forms obtained by the a-c. bridge method and the induction method, as seen on a cathode-ray oscilloscope for a particular circumference of tube *C*. The three large cracks known to be present in the tube at this location are better defined by the induction method than by the a-c. bridge method. The higher definition of the induction method is due to its characteristic sensitivity to the direction of the field. The definition is inversely proportional to the size of the detector. Realizing that increasing the definitive ability of the detector by decreasing its over-all dimensions had a practical limit, a new technique was used. A detector was designed, using the theory of magnetic recording heads, which, by the use of a low permeability gap in the magnetic circuit, concentrates the sensitivity of the detector at the gap (see Fig. 7). This type of detector, when run on tube *C*, showed a marked improvement in defining the known cracks. Increasing the definition of the detector now became a matter of

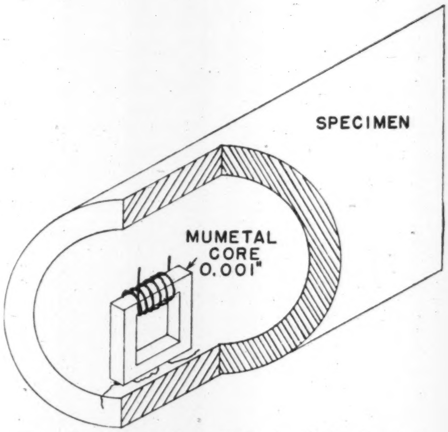

FIG. 5.—Closed Core Detector $\frac{5}{8}$-In. Square.

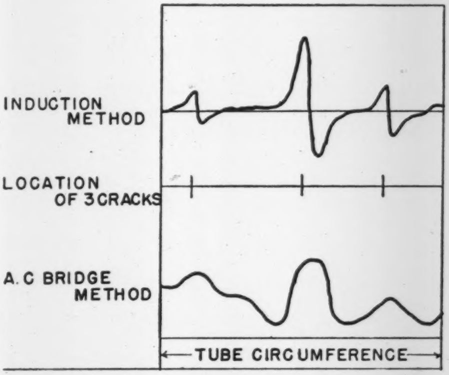

FIG. 6.—A Particular Circumference of Tube *C* as Seen on a Cathode-Ray Oscilloscope by Two Methods.

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[5] R. L. Sanford and Wm. B. Kouwenhoven, "Location of Flaws in Rifle Barrel Steel by Magnetic Analysis," *Proceedings*, Am. Soc. Testing Mats., Vol. XIX, p. 81 (1919).

producing a detector core built to closer tolerances, so as to have a smaller gap, rather than decreasing its over-all dimensions.

The higher definition available simplified the evaluation of data, in so far as cracks close together in the tube could be detected separately. The signals presented on a cathode-ray oscilloscope require a mental integration of the data with time, as well as the interpretation of this information. The same type of interpretation or rearrangement of the data must be made when using a strip chart recorder, which presents a graph showing signal magnitude plotted against time or distance. This type of strip chart recorder has the additional handicap of too high an inertia to reproduce the signals faithfully. Thus, the evaluation of the shape, extent, and location of a crack is difficult and subject to error.

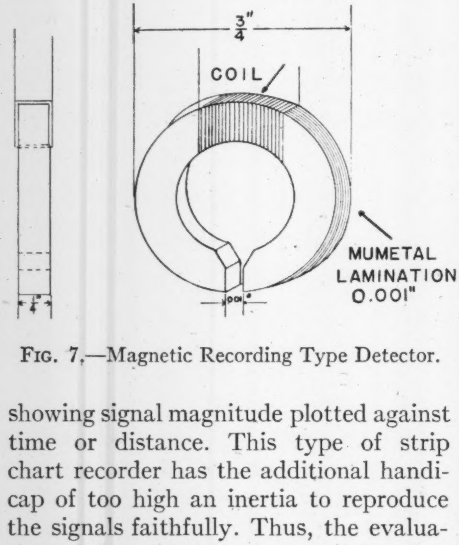


FIG. 7.—Magnetic Recording Type Detector.

Consequently, an investigation of available recording techniques was undertaken with regard to their sensitivity, inertia, cost of operation, and particularly the form of presentation of the data. Investigation of facsimile recorders used in communications work, as suggested by G. Keinath in the discussion of the Hastings paper,[4] revealed a possibility of recording the three dimensions of the flaw at one time. The helix type facsimile recorder, using electrosensitive paper, was selected as the best compromise to meet the criteria.

HELIX FACSIMILE RECORDER

In the helix type of recorder, the recording operation is accomplished by means of a cylindrical drum with a single turn forming the recording electrode or helix. The electrosensitive paper passes between the helix and a narrow-edged strip of metal called the recorder plate. The signal current is passed from the recorder plate through the paper to the helix, or in the reverse direction, thus marking the paper between the recorder plate and helix. In one revolution of the helix drum, the contact area between the helix and the recording plate passes across the width of the paper, and the paper is advanced a distance equal to the width of the electrodes.

The term "electrosensitive" means that the paper is capable of changing color when a current is passed through the paper. The printing takes place on one side of the paper for a given current direction through the paper, and on the reverse side for the opposite current direction. Thus, an a-c. signal may be displayed by using both sides of the chart record. The paper has a threshold voltage for any given writing speed. Printing density is a function of the current passed through the paper, and there is a limit to the amount of power that can be applied to the paper, depending on the amount of moisture present in the paper. Recording takes place under any light condition, without the need for further processing or fixing. The recording is as permanent as the usual printed record.

The presentation of three dimensions is easily accomplished by using the

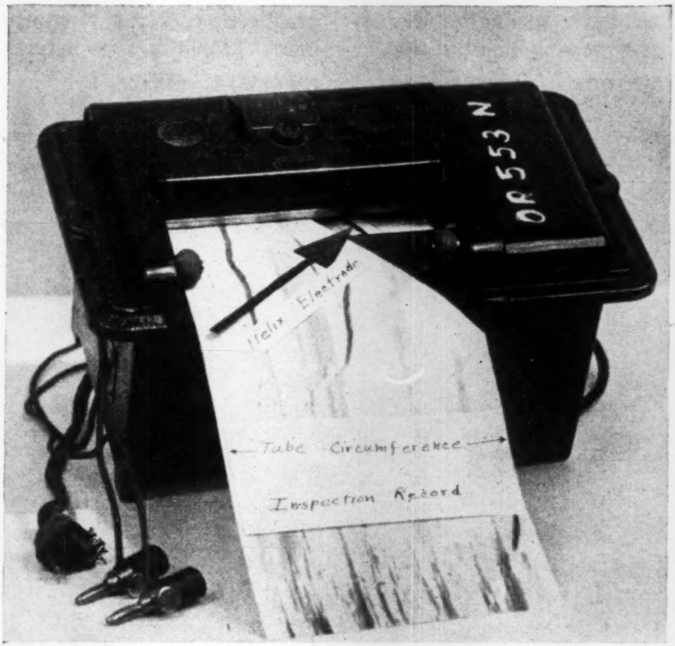

OR 553 N
Helix Electrode
Tube Circumference
Inspection Record

FIG. 8.—Helix Recorder Using Electrosensitive Paper.

density of the marking as one dimension, in addition to the length and width of the record.

After observation of this device in operation, procurement was made of a recorder using a 4-in. wide chart paper with a helix speed of 360 rpm. and a paper advance of 0.010 in. per helix revolution. The scanner mechanism shown in Fig. 4 was properly geared, so that the detector revolved around the circumference of the tube at 360 rpm. By synchronizing the detector with the recorder, the chart width was made to represent the tube circumference, and the chart length the tube length. Thus, the flaws in the tube were displayed in map-like form on the chart record. Each point on the bore surface of the tube was represented by a particular point on the record. Figure 8 shows this recorder with a record of several cracks.

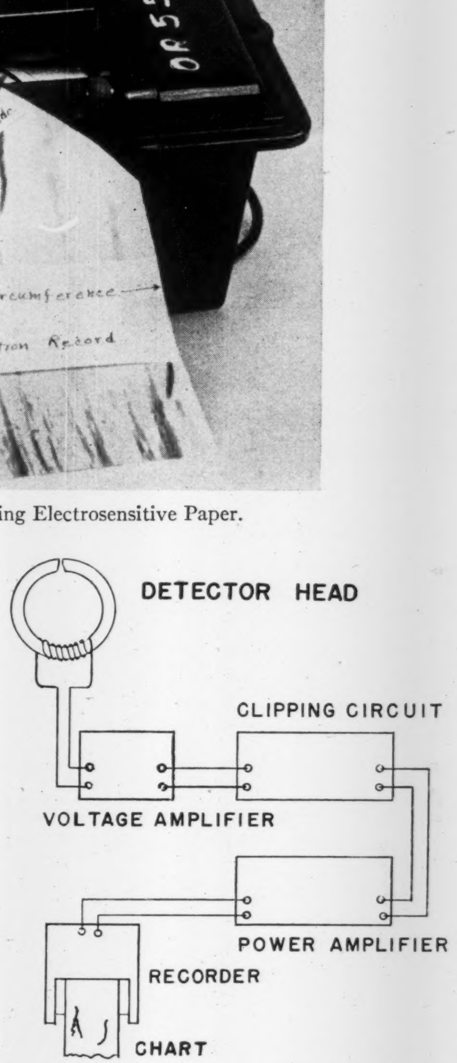
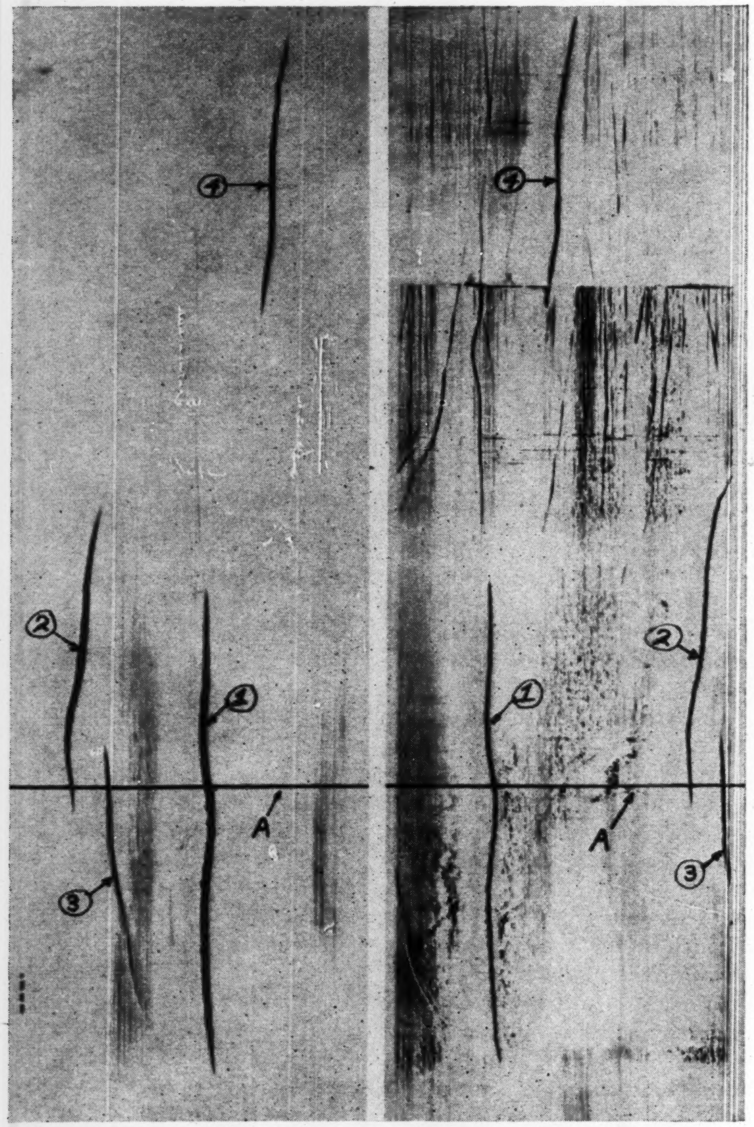

DETECTOR HEAD
CLIPPING CIRCUIT
VOLTAGE AMPLIFIER
POWER AMPLIFIER
RECORDER
CHART

FIG. 9.—Block Diagram of Induction Method Instrumentation with Facsimile Recorder.

Use of the high definition detector and facsimile recorder produced an excellent record of the cracks visible in tube *C*. The latitude of the electrosensitive paper was found to be insufficient to display the range of cracks detectable, and in order to display the smallest cracks, a clipping circuit was built to limit the magnitude of the signals impressed on the recorder. Thus, the smaller cracks could be amplified to



(*a*) Low Sensitivity (*b*) High Sensitivity

FIG. 10.—Inspection Record of Tube *C* by Induction Method at Two Different Levels of Sensitivity. Line A indicates the circumference used in tube *C* to obtain the data in Fig. 6.

the maximum recording level of the paper, without the larger cracks being amplified beyond the capacity of the paper. The complete instrumentation of the induction method is indicated in the block diagram, Fig. 9. Two records of tube *C* are shown for different levels of sensitivity in Figs. 10 (*a*) and (*b*).

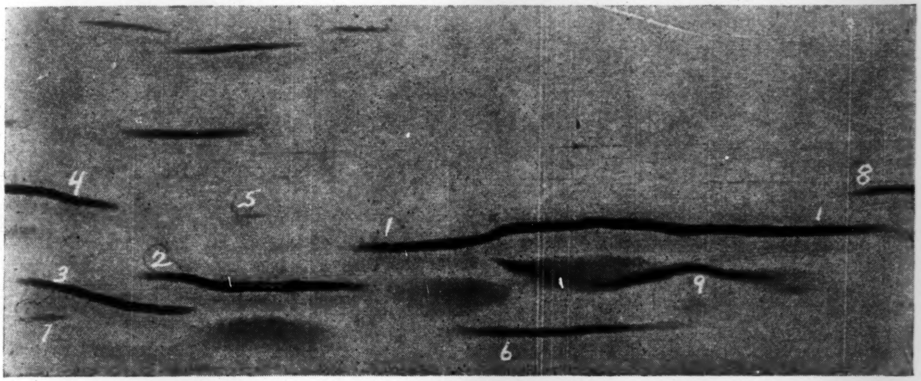


FIG. 11.—Inspection Record of Tube *G* by Induction Method.
Numbers correspond to those in Fig. 13.

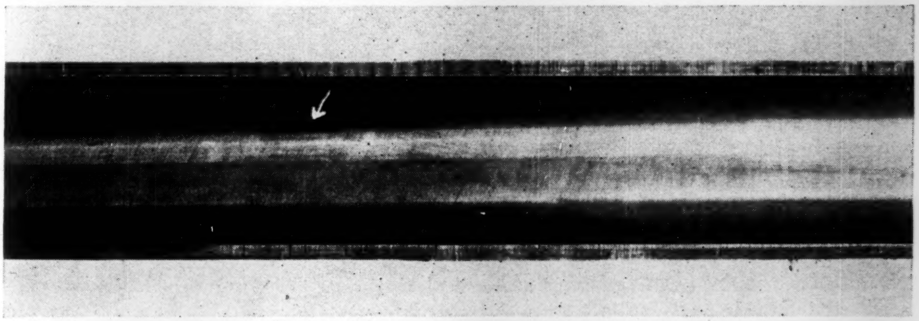


FIG. 12.—Magnetic Particle Test on Inside of Tube *G* Showing One Pattern.

Calibration of the signal magnitude in terms of crack depth has not been undertaken on a rigorous basis. A prerequisite for the calibration of crack depth is the calibration of tube magnetization. This has been accomplished by use of a suspended magnet gaussmeter. The gaussmeter probe was inserted in a clearance hole drilled perpendicular to the wall of the tube near its end and beyond finished dimensions. The reading obtained from the gaussmeter was not an absolute measure of the field level, but was useful as a figure of merit for comparing levels of magnetization and observing changes of the level as the tube was subjected to various processes.

A study of thin-wall tubes by the induction method resulted in three types of data:

1. Those readily recognizable as cracks.
2. Those appearing to be stress concentrations.
3. A class of signals that could not be related to any conditions, detectable by other means, in tubes.

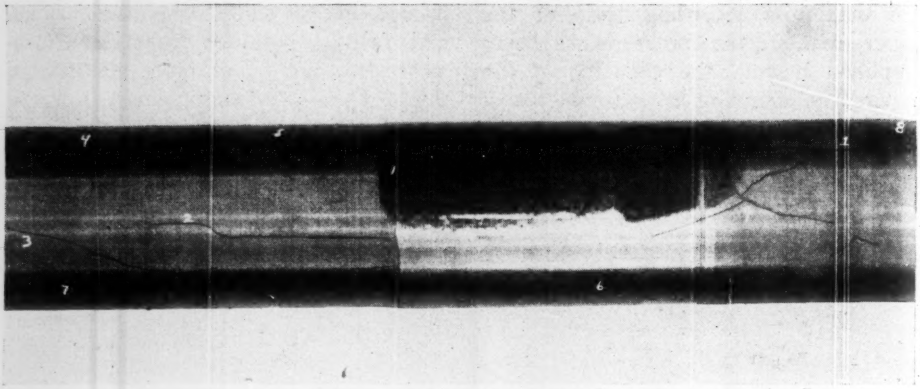
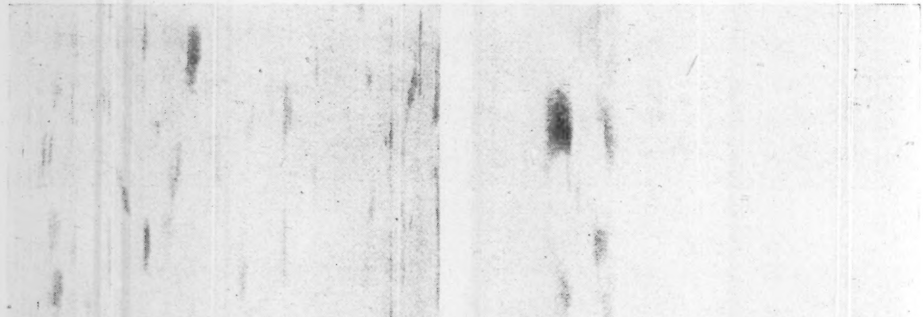


FIG. 13.—Magnetic Particle Test on Outside of Tube *G*.



(*a*) Before Stress Relieving (*b*) After Stress Relieving

FIG. 14.—Inspection Records of Tube *F* by Induction Method.

Tube *G* was representative of the first class of data. The flaw detector record of this tube is presented in Fig. 11, and was made by scanning the bore surface in the usual manner. The tube was cut in half longitudinally and magnetic particle tests performed on the inside and outside surfaces, as shown in Figs. 12 and 13. The magnetic particle test showed that all cracks were open to the outside surface of the tube. The magnetic particle test on the inside could only detect one of these cracks. As can be seen in Fig. 12, the pattern on the inside was not sharply defined, indicating that the corresponding crack was not open to this surface. The flaw detector working from the inside showed all the cracks that the magnetic particle test had been able to detect from the outside. This indicates the high sensitivity of the method. The correlation between cracks seen on the outside of the tube and as recorded from the inside of the tube are displayed by corresponding numbers on Figs. 11 and 13.

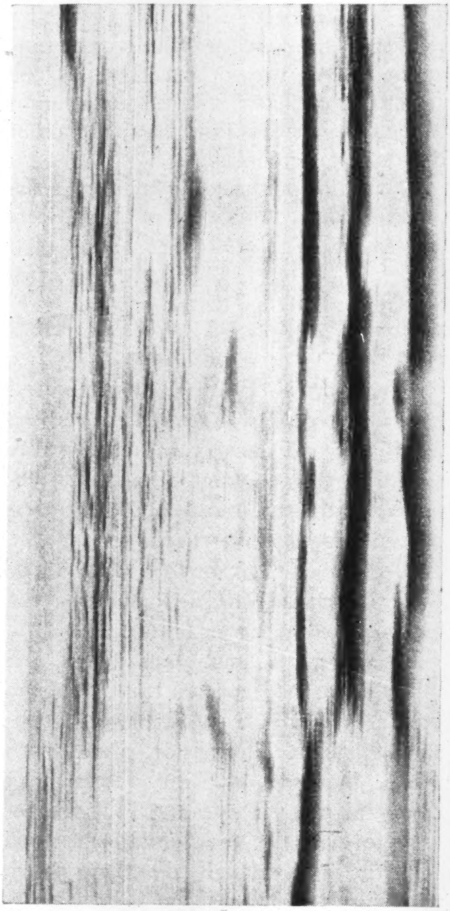


FIG. 15.—Inspection Record of Tube *E* by Induction Method.

The record of tube *F* is presented as an example of what is believed to be the response of the detector to stress concentrations. In particular, it should be noted that the prominent pattern indicated on the original record, Fig. 14(*a*), is not seen in the record of this tube taken after stress relief by heat treatment for 4 hr. at 1100 F. (Figure 14(*b*)).

Tube *E* is typical of the third type of record obtained, as displayed in Fig. 15. Tube *E* was cut at certain sections and studied for cracks, nonmetallic inclusions, and any inhomogeneities or structural variations that might correlate with the flaw detector record. No correlations could be made with the record.

CONCLUSIONS

1. The a-c. bridge method is not considered to be satisfactory for production inspection of tubes for cracks.

2. Cracks which are detectable by visual and magnetic particle test procedures can be reliably recorded and interpreted as such by means of the induction method. By employing higher instrument sensitivity, sub-surface cracks and cracks smaller than those detectable by visual and magnetic particle methods, can be recorded. However, the higher instrument sensitivity permits detection of inhomogeneities other than cracks, which are recorded. Correct interpretation of

records made at high sensitivity levels is dependent upon the complexity of the specimen, a knowledge of its history and the experience of the operator.

3. Because of the following advantages, it is considered that the induction method described in this paper is capable of being engineered into a satisfactory production inspection tool:

(*a*) High speed of operation, combined with reliable performance.
(*b*) Simplicity of operation.
(*c*) High available sensitivity.
(*d*) Good resolution.
(*e*) Non-destructive in nature.
(*f*) Automatic data collection minimizing human error.
(*g*) Permanent record of inspection.
(*h*) Residual magnetism used for test; thus, one magnetization will suffice for a number or all of the inspections after various production stages.

To sum up, the most important advantage over previous methods is the automatic detection and presentation of the data in picture form at a fraction of the time required before.

The author wishes to acknowledge the contributions to this work of C. H. Hastings and S. W. Carter.

DISCUSSION

MR. T. ZUSCHLAG.[1]—Mr. Kodis's paper describes an interesting attempt to find a solution for an intriguing problem of magnetic testing—inspecting the inside of steel tubing by magnetic instrument methods.

I am particularly interested in Mr. Kodis's statement that the magnetic recording head pickup he used had yielded better results than the original approach by means of magnetic probe coils arranged in a permeability bridge combination. Having worked along the same lines, I can easily understand Mr. Kodis's reaction when the performance of the magnetic probes he had been using first proved to be disappointing. However, here I would like to mention that it has been possible to improve this performance by modifying the design of the probes and of the electronic circuits activated by the probes.

The results obtained with the modified magnetic probe arrangements have been much more satisfactory and conceivably might prove superior to the results obtained with the recording head type pickup described in the paper.

It is hoped that further data about these improved magnetic probes can be presented in the near future.

MR. R. D. KODIS (*author*).—It certainly is to be expected that the a-c. bridge method can be utilized for obtaining much information about steel tubes. However, for the detection of cracks at a rapid rate and in a manner easy to evaluate, the induction method with the improvements described herein has many advantages.

Since it has numerous potential applications to existing inspection problems, it will be interesting, as well as profitable, to find a means of overcoming the limitations of the a-c. bridge method.

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[1] Vice President, Magnetic Analysis Corp., Long Island City, N. Y.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON CORROSION OF MATERIALS AT ELEVATED TEMPERATURES

This Symposium on Corrosion of Materials at Elevated Temperatures was held at the Opening Session of the Fifty-third Annual Meeting of the American Society for Testing Materials in Atlantic City, N. J., June 26, 1950. The Gas Turbine Panel of the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals sponsored and developed the Symposium.

The following papers were presented:

Introduction—C. T. Evans, Jr.
Coal Ash Corrosion of Metals at Elevated Temperatures—C. T. Evans, Jr.
Stress Corrosion Tests on Turbo-Supercharger Materials in the Products of Combustion of Leaded Gasoline—G. B. Wilkes, Jr.
Creep as a Surface Dependent Phenomenon—M. R. Pickus and E. R. Parker
The Effect of Environment on the Stress-Rupture Properties of Metals at Elevated Temperatures—O. Cutler Shepard and Willis Schalliol
Preliminary Studies of the Effect of Oxidizing Sulfurous Atmospheres on the Rupture Strengths of Inconel "X" and Inconel—A. M. Talbot and E. N. Skinner
Hydrogenizing Effect of Steam on Ferrous Alloys at Elevated Temperatures—C. A. Zapffe and F. K. Landgraf
Oil Ash Corrosion of Materials at Elevated Temperatures—C. T. Evans, Jr.
The Growing Role of Protective Coatings for Metals in High-Temperature Service—W. N. Harrison

The papers together with discussions are issued as ASTM Special Technical Publication No. 108 entitled, "Symposium on Corrosion of Materials at Elevated Temperatures."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON PLASTICITY AND CREEP OF METALS

The papers in this Symposium on Plasticity and Creep of Metals were presented at the First Pacific Area National Meeting of the American Society for Testing Materials in San Francisco, Calif., October 10–14, 1949.

The following papers were presented:

Introduction—John E. Dorn
The Experimental Exploration of Plastic Flow in Sheet Metals—L. R. Jackson and W. T. Lankford
Forming Parameters and Criteria for Design and Production—William Schroeder
The Use of Creep Data in Design—Howard C. Cross and L. R. Jackson
Super Creep-Resistant Alloys—J. W. Freeman, D. N. Frey, E. E. Reynolds, and A. E. White

These papers together with discussion are issued as ASTM Special Technical Publication No. 107 entitled, "Symposium on Plasticity and Creep of Metals."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON THE NATURE, OCCURRENCE, AND EFFECTS OF SIGMA PHASE

The Symposium on the Nature, Occurrence, and Effects of Sigma Phase was held at the Third and Sixth Sessions of the Fifty-third Annual Meeting of the American Society for Testing Materials in Atlantic City, N. J., June 26–27, 1950.

The two sessions were sponsored by the Research Panel of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals.

The following papers were presented:

- Introduction—Francis B. Foley
- Identification and Mode of Formation and Re-Solution of Sigma Phase in Austenitic Chromium-Nickel Steels—E. J. Dulis and G. V. Smith
- Sigma Phase in Several Cast Austenitic Steels—V. T. Malcolm and S. Low
- X-ray Study of the Sigma Phase in Various Alloy Systems—Pol Duwez and Spencer R. Baen
- Sigma Phase in Chromium-Molybdenum Alloys with Iron or Nickel—John W. Putnam, N. J. Grant, and D. S. Bloom
- The Tetragonality of the Sigma Phase in the Iron-Chromium System—L. Menezes, J. K. Roros, and T. K. Read
- The Formation of Sigma Phase in 17 per cent Chromium Steel—J. J. Heger
- Sigma Phase and Other Effects of Prolonged Heating at Elevated Temperatures on 25 per cent Chromium–20 per cent Nickel Steel—G. N. Emmanuel
- The Occurrence of the Sigma Phase and Its Effect on Certain Properties of Cast Fe-Ni-Cr Alloys—J. H. Jackson
- The Formation of Sigma and Its Influence on the Behavior of Stabilized 18 per cent Chromium-8 per cent Nickel Steels in Concentrated Nitric Acid—Raymond S. Stewart and Stephen F. Urban
- Some Notes on the Structure and Impact Resistance of Columbium-Bearing 18-8 Steels After Exposure to Elevated Temperatures—W. O. Binder
- Observations of the Effect of Sigma on the Mechanical Properties of Columbium-Stabilized Weldments in Austenitic Stainless Steels—F. W. Schmitz and M. A. Scheil

These papers together with discussions are issued as ASTM Special Technical Publication No. 110 entitled, "Symposium on the Nature, Occurrence, and Effects of Sigma Phase."

WHY TYPE II CEMENT?*

By F. H. Jackson[1]

Synopsis

The paragraph which defines the five types of portland cement covered by the A.S.T.M. Standard Specifications for Portland Cement, C 150–49,[2] states (1) that type I cement is "For use in general concrete construction when the special properties specified for types II, III, IV, and V are not required" and (2) that type II cement is "For use in general concrete construction exposed to moderate sulfate action or where moderate heat of hydration is required." In spite of this limitation in scope there has been a marked tendency on the part of many consumers to consider type II as an improved general purpose cement, particularly with respect to resistance to severe natural weathering (freezing and thawing). The paper discusses the reasons for this trend and also traces briefly the history of the development of cement specifications in this country. The comparative performance of eight typical type I and five typical type II cements in four of the projects of the "Long-Time Study of Cement Performance in Concrete" which have been exposed to severe natural weathering for periods up to 8 years, is discussed. Observations of the condition of the concrete structures in these four projects indicate that, up to the present time, there is little choice between the two types as regards the influence of cement upon durability. In fact, greater differences were found between individual cements of both types than between the average values for each type. These facts would indicate that, in so far as the present indications of the Long-Time Study are concerned, type II cement is not the answer to the problem of durability of concrete as affected by the cement. The paper concludes with the statement that further research is necessary to determine to what extent variations in cement properties outside of the range included in the present Long-Time Study program, may affect durability.

Ever since the adoption by this Society in 1941 of the so-called five-type specification for portland cement, many users as well as manufacturers of cement have been raising the question indicated by the title of this paper, "Why Type II Cement?" Does this particular type supply a need that cannot be met by one of the other four types recognized in A.S.T.M. Specification C 150? Specifically, can it be justified as a "modified" or "improved" type I cement for important general purpose construction? The demand for an answer to this question is becoming more and more pressing. For example, the current shortage of portland cement has emphasized the importance of the question from the economic point of view. Manufacturers are questioning the insistence of certain consumers that a type II cement be furnished for use under conditions where the A.S.T.M. definitions indicate that type I is adequate. It seems desirable to survey this entire matter at this time to determine to what extent it may be possible to supply an answer from information currently available.

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* This paper was presented at the Session on Cement and Concrete, held at the First Pacific Area National Meeting of the Society, San Francisco, Calif., October 10–14, 1949.

[1] Principal Engineer of Tests, Physical Research Branch, Bureau of Public Roads, Washington, D. C.

[2] 1949 Book of A.S.T.M. Standards, Part 3, p. 1.

Unlike type III, high-early-strength cement, type IV, low-heat cement, and type V, sulfate-resisting cement, all of which are definitely special purpose cements, as indicated by their names, type II occupies a sort of no-man's land between the general purpose type I and the three special types. The description of type II as given in A.S.T.M. Specification C 150 states that this type is suitable for use under conditions where moderate sulfate resistance or moderate heat of hardening is required. Furthermore, the description of type I states that this type is "for use in general concrete construction when the special properties specified for types II, III, IV, and V are not required." In spite of these limitations in scope, there has been a definite tendency on the part of many consumers to consider type II as an improved general purpose cement and to specify it on work under conditions where neither the heat of hardening nor sulfate resistance may be considered as important in themselves. A number of factors have apparently contributed to this situation.

In the first place, there has been for many years a well-defined feeling among certain consumer groups that restrictions in chemical composition along the lines specified for type II cement will insure more weather-resistant concrete than would be possible under the much broader type I specification. The requirements which are usually considered most significant are, first, the limitation of not to exceed 8 per cent tricalcium aluminate (C_3A) and, second, the limitation of not to exceed 50 per cent tricalcium silicate (C_3S). Engineers differ as to the relative importance of these two limitations. For example, one state highway department sets a limit of 50 per cent C_3S but has no limit for C_3A. Another highway department limits the C_3A to 8 per cent but lets the C_3S go to 55 per cent. Others still further modify the specification by additional requirements, all designed to produce a more durable cement. It should be stated, however, that, so far as the state highway departments are concerned, most of them continue to specify type I in their general construction program.

The demand for type II cement for general concrete work has doubtless been encouraged also by the fact that many Government agencies which are large users of cement, including the Corps of Engineers, the Bureau of Reclamation and the Tennessee Valley Authority, have for years favored this type for important general purpose construction. The Bureau of Reclamation, in particular, has favored type II in all but small structures and maintenance work. The Bureau prefers the modified type II because the low C_3A content results in smaller volume changes whereas the percentages of C_3S and C_2S are sufficiently high to insure good strength. An additional advantage, according to the Bureau, is that this type is usually ground somewhat finer than type I, resulting in less bleeding, better workability, and more impermeable concrete.[3]

The demand for type II cement has been encouraged also by certain theoretical considerations and test indications which will be discussed later. It has been still further encouraged by the description of type II on Federal Specification SS-C-192, which reads as follows: "For use in general concrete *construction and in* construction exposed to moderate sulfate action, or where moderate heat of hydration is required." The italicized words do not appear in the corresponding A.S.T.M. specification. Clearly the inference conveyed by the Federal Specification is that there is a place for type II as a general purpose cement.

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[3] Concrete Manual, U. S. Bureau of Reclamation, Denver, Colorado, 4th Ed., 1942.

There is also the question of uniformity. Many users of portland cement have felt that the lack of chemical limitations, other than the time-honored limits on sulfur trioxide, magnesia and insoluble residue, permit of too wide a variation in compound composition and that certain restrictions such as those shown for type II, are desirable from this point of view alone if for no other reason.

It is probable that in the case of many consumers the preference for type II as an all purpose cement has been developed more as the result of long-time experience in the field than on the basis of carefully planned and well executed research programs. However, the fact remains that it has been used for a long time with highly satisfactory results and the question naturally arises, is this good performance record due primarily to the cement itself, or to other factors? In other words, would type I cement have served just as well under the same circumstances? It was for the purpose of answering this, as well as other questions along the same line, that the Cement Industry about ten years ago initiated the project for the long-time study of the performance of cement in concrete.[3] The results of this most interesting and important project are just now becoming available for study. These results will be discussed in the present paper only to the extent to which they bear upon the particular subject under discussion. The over-all picture is presented in a paper by G. J. Verbeck.[4]

It is proposed in this paper to discuss the need for a type II cement primarily from the standpoint of whether this type should be considered as an improved type I for use in concrete exposed to severe natural weathering. The history of the development of cement specifications in this country, particularly the part played by the Society in this work, will be outlined briefly. Considerations which led to the adoption of the type II specification will be discussed, including the original thought, held by a substantial consumer group on A.S.T.M. Committee C-1 on Cement, that the description for this type should have included reference to its superior weather resistance. The results of laboratory investigations will be discussed briefly. Finally, reference will be made to the comparative performance to date of the two types in four of the Long-Time Study projects which have been subjected to severe natural weathering.

HISTORICAL

As would be expected, the first attempt to standardize cement specifications in this country was along the lines of developing uniform test methods. In 1885, the Committee on Uniform Tests of Cement of the American Society of Civil Engineers issued a report in which it recommended a series of standard procedures for testing cement, both natural and portland. Prior to 1904 there seems to have been little or no attempt made to standardize specifications, with the result that everyone wrote his own requirements and literally hundreds of specifications, no two alike, began to appear. Furthermore, the original recommendations of the A.S.C.E. regarding test methods appear to have found less and less acceptance as time went on, with the result that numerous modifications of the original procedures came into current use, still further complicating the specification picture. According to Mr. Robert W. Lesley, from whose interesting book "History of the Portland

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[4] George J. Verbeck and Cecil W. Foster, 'Long-Time Study of Cement Performance in Concrete—Chapter 6—The Heats of Hydration of the Cements, see p. 1235 this publication.

Cement Industry in the United States" these brief historical notes are taken, the manufacturers struggled against this situation until around the close of the century, when efforts were again made to bring some order out of the chaos of confusing and sometimes conflicting specifications then in current use.

In 1903, the newly organized American Society for Testing Materials appointed a committee to work on specifications for portland cement as well as on methods of testing. This committee has functioned ever since as the principal specification writing body for portland cement in this country. Unlike the original A.S.C.E. committee which, with one exception, was made up entirely of engineers representing consumer groups, the newly organized A.S.T.M. committee was composed of representatives of both producer and consumer interests. This, of course, is the principle under which our Society functions; that is, the principle of equal and joint responsibility of both manufacturers and users in the preparation of standards. By and large, the principle has proved thoroughly satisfactory, as the wide recognition given our standards both in this country and abroad will attest.

From the first, the Cement Committee of the A.S.T.M. concentrated on the preparation of a single standard specification for cement. It was confronted with the immediate problem of reconciling, in so far as possible, the multitude of specifications in use at that time in such a way as to secure general acceptance. Its first efforts in this direction resulted in the standard specification of 1904, the first A.S.T.M. specification for portland cement. This specification appears to have had rather general acceptance from all users except the Government. However, further effort to correlate the A.S.T.M. and the Government specifications led, in 1916, to a revision in which most of the differences were reconciled. Finally, in 1921, the specification was still further revised to effect complete agreement and to constitute in the words of Mr. Lesley ". . . an agreement between all authorities in the United States having to do with a single uniform specification."

Thus it will be seen that the problem which was faced by the Industry in 1900—that of satisfying the demands of a multitude of specification writers—had been solved by 1921 through the virtually unanimous acceptance of a single specification for a general all-purpose cement of standard quality. This single standard remained in effect, with but one break, until 1941 when the present five-type specification was adopted. The break came in 1936 with the adoption of a specification for high-early-strength cement, a development which came about largely as the result of consumer demand for such cement for use in road construction where early opening to traffic was demanded. However, long before this the pendulum had again begun to swing back in the direction of multiple specifications. The Bureau of Reclamation had for some time been using "low-heat" and "moderate-heat" cements for mass concrete and had developed specifications for these cements. Federal specifications for moderate-heat and sulfate-resisting cements had been developed, and the Board of Water Supply of New York City, under the guidance of Mr. Thaddeus Merriman, had adopted its unique specifications covering control of manufacturing operations.

Meanwhile, research investigations, notably those conducted by Bogue under the Portland Cement Assn. Fellowship at the National Bureau of Standards, and by the Association itself in Chicago, were pointing to the possible justification for additional chemical limits to control compound composition.[5] Users began more and more to question the "single-standard" philosophy of Committee C-1, with the result that, in 1938 the chairman, Mr. P. H. Bates, appointed a special committee consisting of ten consumer and general interest members to study and report on the question "How Many Specifications for Portland Cement?" C. H. Scholer, of the Kansas State Agricultural College, who was the first chairman of this group, resigned in the spring of 1939, and the author was named chairman. The special committee, after much correspondence and several meetings, finally recommended a single specification covering five types of cement patterned largely after the Federal Specifications. The description of the types and the various chemical and physical test limits which were proposed were later adopted by Committee C-1 with very few changes except with respect to the scope of type II. The original description of this type as prepared by the special committee read as follows:

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[5] H. F. Gonnerman, "Study of Cement Composition in Relation to Strength, Length Changes, Resistance to Sulphate Waters and to Freezing and Thawing of Mortars and Concretes," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 244 (1934).

> *Type II.*—For use in concrete exposed to weathering or where a cement having a moderate heat of hardening or moderate sulfate resistance is required.

The reference to weather resistance in the above description had been inserted by the special committee on the basis of various considerations, including favorable service records shown by this type as well as the result of trends revealed by laboratory investigations regarding the effect of variations in the compound C_3A on volume constancy and durability-trends which at that time seemed to the special committee to be quite significant. For example, the special committee had available for reference a report issued in 1936 by the U. S. Bureau of Reclamation entitled "Special Cements for Mass Concrete." This report contained a summary of the results of a number of independently conducted laboratory investigations on the influence of compound composition on the properties of concrete. Although most of these conclusions applied primarily to requirements for cement for use in mass concrete (type IV) there were several which related directly to the matter of durability. It will be of interest to review these conclusions in the light of the discussion to follow:

> "(1) Compound composition and fineness are outstanding factors affecting the properties of cement and the resulting concrete and constitute practical means for controlling cement according to desired characteristics.
>
> "(2) The major cement compounds, C_3S, C_2S, C_3A, and C_4AF, have definite effects on compressive strength, heat of hydration, durability, and other properties of cement and concrete. C_3A is definitely shown to be the least desirable compound in portland cement and C_3S and C_2S are the most desirable constituents.
>
> "(5) Tricalcium aluminate usually increases the strength somewhat at ages of 7 and 28 days, but at later ages it is detrimental.
>
> "(13) Modified cements give satisfactory strengths at both early and later ages. They . . . seem to be better suited for general purposes than the low heat and standard types.
>
> "(22) Results of these tests performed by a number of independent investigators show very definitely that mortars and concretes containing cements of high C_3A content offer poor resistance to freezing and thawing."

From the above it would seem that, at the time the five-type specifications were written, there was considerable justification for the desire of the special committee to recognize the superior weather-resistant properties of type II portland cement. On the other hand, the full membership of Committee C-1, including of course the producers, felt that, in view of the long-time favorable record of many type I cements in the matter of durability, this type should not be discriminated against as indicated by the original scope. The specification as finally approved, therefore, limited the recommended uses for type II to conditions where moderate-heat of hardening and moderate sulfate resistance were required.

Discussion of the effect of compound composition on durability should not be confined to work done prior to 1940. The studies by Lerch on the effect of variations in SO_3 content of cements, as published in 1946,[6] have a direct bearing on this problem. The earlier work was all done on cements having a constant SO_3 content within the then existing limits of the specifications. Lerch has shown that the strength and volume constancy of mortars containing cements of varying C_3A content can be influenced appreciably by varying the amount of gypsum added for the purpose of retarding the set. In general, he found that the optimum SO_3 content increased with the amount of C_3A in the cement and that, for many cements, this optimum exceeded the maximum percentage allowed in the specifications. Lerch's investigations showed also that, for cements of the same C_3A content, those high in alkalies require more gypsum for optimum results than those low in alkalies. His work gives a possible explanation of why certain type I cements have not proved as durable as expected and also suggests a solution.

Work on SO_3 content is being actively continued by a subcommittee of Committee C-1 under the chairmanship of H. S. Meissner, of the Bureau of Reclamation, who is working both on the problem of developing a standard method for determining the optimum SO_3 content and that of developing further information regarding these relationships, particularly the effect of proper retardation on resistance to freezing and thawing. In the meantime, Committee C-1, recognizing the validity of the relations indicated by Lerch, has modified its specifications for both type I and type III cement by raising the maximum permissible limit for SO_3, five-tenths percentage points in each case, making the limit for type I, 2.5 per cent, and the limit for type III, 3.0 per cent (the higher limit for both types to apply only when the C_3A content exceeds 8 per cent).

INDICATIONS OF THE LONG-TIME STUDY

The first four chapters of the report of the "Long-Time Study of Cement Performance in Concrete" including the history and scope of the investigation, the manufacture of the test cements, the chemical and physical properties of the cements, and the microscopical study of the clinkers have been published both in the *Journal* of the American Concrete Inst. and by the Portland Cement Assn.[7] Therefore the project will be described in this paper only to the extent necessary to provide a background for the discussion to follow.

The project was first suggested about ten years ago by P. H. Bates, at that time Chief of the Division of Clay and Silicate Products of the National Bureau of Standards, and also Chairman of Committee C-1 on Cement of the A.S.T.M. It will be recalled that it was at just about this time that Committee C-1 adopted the five-type specification and many discussions were in progress, both in and outside of the committee, as to the value of chemical limitations, manufacturing control, and other special requirements. Mr. Bates felt that the best way to settle these arguments would be to build a series of full-size structures in various parts of the country with a number of cements meeting the requirements of the different types recognized in Specification C 150, and to observe the performance of these structures, particularly with respect to durability, throughout the years. The cements were to be made by the usual methods but complete records of the manufacturing details, including burning, cooling and grinding, were to be kept. Complete chemical and physical tests were also to be made including determination by every known test, special as well as standard.

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[6] William Lerch, "The Influence of Gypsum on the Hydration and Properties of Portland-Cement Pastes," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1252 (1946).

[7] *Journal*, Am. Concrete Inst., Vol. 19, Nos. 6, 7, 8, and 9, February, March, April, May 1948; Research Laboratories, Portland Cement Assn., Chicago, Ill., *Bulletin 26*, August 1948.

Early in 1940 Mr. Bates' proposal was accepted by the Portland Cement Assn. A 12-man Advisory Committee (eight from consumer groups including several U. S. Government agencies, and four from the Industry) was organized to set up a program and the sum of $386,100 was appropriated by the Association to finance it. The complete program for the investigation as finally adopted by the Association upon recommendation of the Advisory Committee involved the manufacture of 27 different cements to be used in several types of job-size and near job-size structures located in various parts of the country. Twenty-one of these cements were of the normal, nonair-entraining variety in general use at that time, classified as follows: Eight type I, five type II, three type III, four type IV and one type V. In addition, six air-entraining cements were made by intergrinding flake Vinsol resin with the same clinkers as were used in the manufacture of the corresponding plain cements. Four of these were type I, one type II and one type III. These are identified in the discussion and tables to follow by the letter T following the cement number. The first numeral in each cement number identifies the type. For example, cement 12 is a type I, nonair-entraining cement, cement 24 is a type II, nonair-entraining cement and cement 12 T is a type I, air-entraining cement made from the same clinker as was used in the manufacture of cement 12.

With certain exceptions, at least 24 of these 17 cements were used in each of the following job-size or near job-size structures: Three experimental roads, one located in western New York, one in western South Carolina, and one in central Missouri; four installations of reinforced concrete piles—one in the fresh water of the Hudson River at Saugerties, N. Y., one in the Atlantic Ocean at Cape Cod, Mass., one in the Atlantic Ocean near St. Augustine, Fla., and one in the Pacific Ocean at Newport Beach, Calif.; the parapet wall of an earth-filled dam at Green Mountain, Colo. (elevation 8000 ft.); the walkway slabs of a multiple arch concrete dam at Florence Lake, Calif. (elevation 7300 ft.); test specimens subject to the action of alkali soils at Sacramento, Calif.; and two so-called experimental "farms" for the study of near job-size concrete, one located at Naperville, Ill., and the other near Perry, Ga.

Of these twelve projects there are four which, by virtue of severe exposure to natural weathering, lend themselves to a study of durability (resistance to alternate freezing and thawing). These are the test roads in New York, the Green Mountain and Florence Lake Dams and the test farm at Naperville, Ill. The test road was built during the late summer and early fall of 1942, the parapet wall of the Green Mountain Dam in the spring of 1943, the walkway slabs at Florence Lake in 1942 and the test farm in the fall of 1941. It is believed that a brief progress report on the present condition of these structures will be of interest. The discussion will be limited to those sections of each project which involve the types I and II cements and their air-entraining counterparts, 18 cements in all.

The New York Test Road:

This pavement is located in New York on State Route 17, between the towns of Wellsville and Bolivar. It is in a region of heavy snowfall with wide and rapid changes in temperature. The location is considered typical of severe winter exposure in this country. The test road is 2.5 miles long and 24 ft. in width. The pavement has an 8–7–8 in. cross-section, with longitudinal joint and transverse expansion joints 1 in. in width spaced 75 ft. apart. Construction was half width (single lane) and all concrete was placed on a 6-in. rolled gravel base. The aggregates were sand and gravel meeting the requirements of the New York Highway Dept. Mix proportions and construction operations were all in accordance with New York State Standards. The cement content was maintained reasonably constant at 6.4 bags per cu. yd. Water contents varied somewhat with the different cements in order to maintain a reasonably uniform consistency from section to section. The pavement was cured with cotton mats kept saturated and in place for 72 hr.

The entire project was divided into three parts, Projects 1, 1A and 1B. Project 1, using 21 plain and 3 air-entraining cements, was intended to be representative of normal concrete paving practice in New York State. It was located on a portion of roadway having slight grades so as to insure that salt would not be used for ice control. Project 1A contained three plain and three air-entraining cements in concrete similar to that of Project 1 except that the slump of the concrete was increased from the nominal 3 in. of Project 1 to a nominal slump of 5 in. Project 1B contained six plain and six air-entraining cements in concrete of the same mix proportions and slump as Project 1. It was located on a section of road where conditions were such that salting would ordinarily be required for ice control. The arrangement made it possible to study cement performance under different conditions of construction and exposure. In all three projects, a local cement was used to start each day's operations.

Each test cement in each project was used four times in pavement sections 150 ft. in length. Inasmuch as the pavement slabs were 75 ft. long, this made it possible to study the behavior of the concrete at an expansion joint located in the center of each test section, a very desirable feature, due to the tendency for disintegration to start at such locations. The construction schedule was laid out so that placing of the four sections containing each cement was separated by as much time as possible without interfering with construction progress or an orderly arrangement of sections in the pavement. This provided a range in placing conditions for each cement.

As a rule concrete pavements located within the area represented by the New York test road (that is, northwestern Pennsylvania and western New York) have not performed any too well from the standpoint of durability. Winters are severe and fluctuations in temperature wide and frequent. It requires the very best in concrete to stand up under such conditions. Experience has indicated also that in the case of pavements which fail prematurely due to lack of durability, the first evidence of distress usually takes the form of D-cracks along the transverse and longitudinal joints and along structural cracks and free edges of the pavement. Furthermore, it has been the author's experience that, in the case of potentially nondurable concrete, these cracks usually become evident within 5 or 6 years and that if they do not form within this period the chances are that the concrete will be durable during the period of the economic life of the pavement. If this is true, the New York test road, which was built in 1942 and has therefore gone through seven winters, is old enough to permit at least tentative comparisons with respect to durability.

The experimental pavement has of course been inspected periodically by members of the Long-Time Study Committee and complete notes have been made of the various sections. An official progress report which it is hoped will be available within the next few months is now in course of preparation. The author visited this project in June of this year (1949) in company with Mr. T. E. Coultry of the Division Office of the Bureau of Public Roads and made a rather detailed examination of the road from the standpoint primarily of evidence of premature weathering as indicated by the formation of D-cracks.

In Table I there have been tabulated for each cement the total number of 75-ft. slabs cast and the number of slabs showing any evidence of D-cracking as determined by this examination. Sections in Project 1A which involved the 5-in. slump concrete with cements 12, 16, and 21 and cements 12 T, 16 T, and 21 T were included in this tabulation. This seemed to be justified, due to the fact that no relation whatever could be traced between consistency and D-cracking. Cement 21, for example, had the same number of D-cracked slabs on Project 1 as on Project 1A.

These data reveal several interesting points. In the first place it will be noted that none of the 48 slabs containing the air-entrained concrete show any evidence of D-cracking after 7 years' exposure. On the other hand, 8 out of 80 slabs containing the plain or nonair-entraining type I cements, or 10 per cent, show D-cracks and 14 out of 48 slabs containing the plain type II cements, or 29 per cent, show evidences of such cracking. Three of the type I cements show no D-cracking, whereas none of the type II cements are entirely free from this defect. It is also quite interesting to note that the two cements which show the greatest amount of D-cracking, without air entrainment, cements 16 and 21, are entirely free from D-cracking in the sections containing the air-entraining counterparts of these cements. The author sees nothing in these data to indicate that the type II cements as a group are performing any better after 7 years' exposure than the corresponding group of type I cements. Furthermore, omitting cement 21, there seems to be little to justify any choice between any of the cements in these two groups. In view of the record to date, it is believed that the ultimate failure of this road will probably be caused by structural breakdown due to movement of heavy loads over unprotected joints and cracks rather than any failure due to lack of durability of the concrete. As a matter of fact, this structural breakdown has already started and will no doubt be slowly progressive due

TABLE I.—PROJECTS 1 AND 1A, NEW YORK TEST ROAD. CONDITION OF PAVEMENT SLABS WITH RESPECT TO EVIDENCE OF ACCELERATED WEATHERING (D-CRACKS).[a]

(Ratings as of June 1949. Road constructed 1942.)

Cement	Number of 75-ft. Slabs	Slabs Showing D-Cracks
No. 11	8	0
No. 12	16	1
No. 13	8	1
No. 14	8	0
No. 15	8	2
No. 16	16	3
No. 17	8	0
No. 18	8	1
Total Type I	80	8
No. 21	16	10
No. 22	8	1
No. 23	8	1
No. 24	8	1
No. 25	8	1
Total Type II	48	14
No. 12 T	16	0
No. 16 T	16	0
No. 21 T	16	0
Total	48	0

[a] D-cracks are defined as fine, parallel cracks, usually filled with a dark-colored deposit, probably calcium carbonate, which form along the edges of the joints and structural cracks, and sometimes along the free edges of the slabs. They are usually considered to be evidence of accelerated weathering leading sometimes but not always to ultimate disintegration. Defects of this type were the only ones considered in preparing the above table.

to the heavy loads which are being carried by this through state highway.

A very interesting section of the New York test road is Project 1B at the east end. This section is located on a 6 per cent grade. Twelve of the test cements, six plain and six containing flake Vinsol resin interground with the same clinkers, were used on this section. It was expected that salt would be used for ice control on this grade and the records of the New York State Highway Dept. show that salt in varying quantities has been used each winter since the construction of the road in 1942.

TABLE II.—PROJECT 1B, NEW YORK TEST ROAD. CONDITION OF SALTED SECTION WITH RESPECT TO SURFACE SCALING.
(Ratings as of June 1949. Road constructed in 1942.)

Cement	Number of 75-ft. Slabs	Slabs Showing Scale		
		Number Showing Scale	Per cent Area Scaled	
			Maximum	Average
No. 11	8	7	35	9
No. 12	8	6	50	23
No. 16	8	8	40	11
No. 18	8	8	20	8
No. 21	8	6	15	7
No. 33	8	4	25	16
Total	48	39	..	12 (Avg.)
No. 11 T	8	1	Trace	0[a]
No. 12 T	8	0	0	0
No. 16 T	8	0	0	0
No. 18 T	8	0	0	0
No. 21 T	8	0	0	0
No. 33 T	8	0	0	0
Total	48	1	Trace	0[a]

[a] Except for a small amount of very light scale or surface wear, the slabs containing the air-entraining cements are entirely free from scale. Slabs rated with respect to scaling only.

Table II shows the amount of surface scaling that has taken place on Project 1B, as revealed by the inspection of June, 1949. It will be noted that surface scale has developed on 39 out of the 48 slabs containing the plain or untreated cements, whereas only one slab out of the corresponding 48 slabs of air-entrained concrete shows any scale. The amount of area scaled on any one slab varies from a trace on one of the slabs containing cement 11T to 50 per cent on one of the slabs containing cement 12. These results reveal very definitely the value of air entrainment in the prevention of surface scale due to the use of ice control chemicals.

The Green Mountain Dam:

The parapet wall built along the crest of this dam is also of interest in connection with our study of the relative durability of type I and type II cements. This project was constructed in 1943 under the supervision of the U. S. Bureau of Reclamation which has filed with the Long-Time Study Committee a very complete report giving not only a detailed description of the condition of the various test panels at the end of 3 years' severe exposure at an elevation of 8000 ft. but also the results of a very comprehensive series of laboratory tests made with the Long-Time Study cements. Twenty-four of these cements were used in the parapet wall. They were used four times in panels 9 ft. long, 4 ft. high and 16 in. in width. One-half inch absorptive form lining was used on the down-stream face of each panel while the upstream face was cast against plywood. The cement content averaged slightly less than 6 sacks per cu. yd., and the water-cement ratio slightly less than 0.6 by weight. The slump of the concrete was approximately 3 in. The conclusions reached by the Bureau of Reclamation as the result of a detailed inspection made after 3 years' exposure were (1) that there was no certainty that any of the rather minor differences then existing were significant as regards the long-time performance of the various cements, and (2) that measurements of length had not demonstrated any significant differences in the behavior of the various cements nor had any unusual expansions been noted. This latter conclusion is quite significant in view of the fact that tests made by the laboratory of the Bureau of Reclamation have shown that the aggregates used in the Green Mountain Dam are reactive with high-alkali cements. Certain of the Long-Time Study cements contained relatively high percentages of alkali.

A rather hasty examination of the concrete test panels made by the author in June 1949 would indicate that no differences have yet developed which could be considered as significant from the standpoint of the comparative performance of the various types of cement. Considerable surface checking and some cracking have developed, but there is little evidence of D-cracking on the test panels themselves. Here again there are evidences that there may be greater differences between particular cements of either type than there are between the average of one type as compared to the others.

Florence Lake Dam:

This dam is part of a hydroelectric development serving southern California and is located in the High Sierras at an elevation of approximately 7300 ft. The climate is very severe and the location may be considered an excellent one for studying durability. The test cements were used in walkway slabs 3 ft. wide and 10 ft. long, each cement being used three times. Along with the walkway slabs, test beams were also cast for exposure to the weather in this same location.

Periodic inspections have been made and an official progress report will be issued. The last official inspection was made in 1948, after 6 years' exposure, by a party representing the Long-Time Study Committee. The results of this inspection are shown in Table III. Reference to the table will show that 3 of the 24 walkway slabs containing the type I cements show evidences of weathering while 7 of the 15 slabs containing the type II cements are similarly affected. The corresponding test beams show 10 out of 32 are affected in the case of type I and 8 out of 20 are affected in the case of type II. However, the most significant difference is again the superior resistance of the air-entrained concrete. In 15 walkway slabs and 20 test beams, none so far show any evidence of distress. Again, as in the case of the New York test road, we find some differences in cements within each type.

TABLE III.—PROJECT 9A. FLORENCE LAKE DAM. CONDITION OF WALKWAY SLABS AND BEAMS.

(Ratings as of 1948. Slabs and beams cast in 1942.)

Cement	Walkway Slabs		Test Beams	
	Number Cast	Number Affected	Number Cast	Number Affected
No. 11	3	0	4	1
No. 12	3	0	4	1
No. 13	3	2	4	3
No. 14	3	0	4	0
No. 15	3	0	4	0
No. 16	3	1	4	0
No. 17	3	0	4	1
No. 18	3	0	4	4
Total Type I	24	3	32	10
No. 21	3	3	4	4
No. 22	3	2	4	4
No. 23	3	1	4	0
No. 24	3	0	4	0
No. 25	3	1	4	0
Total Type II	15	7	20	8
No. 11 T	3	0	4	0
No. 12 T	3	0	4	0
No. 16 T	3	0	4	0
No. 18 T	3	0	4	0
No. 21 T	3	0	4	0
Total	15	0	20	0

Six of the type I cements show no evidence of distress in the walkway slabs as against one of the type II cements. Two of type I cements and one type II cement show no evidence of distress in either the walkway slabs or the test beams. These data certainly give no indication that the type II cements used in this study have so far proved superior to type I from the standpoint of durability. In fact, as in the case of the New York test road, the evidence on its face would indicate a tendency in just the opposite direction.

Experimental Farm at Naperville, Ill.:

In many respects, this is the most interesting of all of the Long-Time Study projects. Due to the relatively small size of the test units it was possible to introduce other variables in addition to cements. These included cement content, aggregate quality, consistency of concrete and methods of placing. Three types of test specimen were made: flat slabs, 30 by 42 by 6 in. in size, cast on the ground; hollow boxes 30 in. square and 30 in. in height, with walls 4 in. thick tapering to 12 in. at the base; and 8 by 8 in. vertical columns 60 in. high. For the flat slabs, three concrete mixes, 5 and 6½ sacks per cu. yd., 3-in. slump and 6½ sacks per cu. yd., 8-in. slump, and three aggregate combinations were used with each cement, making a total of 243 combinations. For the boxes and columns, two cement contents, 4½ and 6 sacks per cu. yd., two aggregate combinations and three consistencies, 1½-in. slump, vibrated, and 3-in. and 8-in. slump, hand placed, were used with each cement, making a total of 324 combinations. This provided an opportunity to compare the performance of the various cements under a wide variety of conditions as well as the opportunity to study the effects of these other variables either for a given cement or for the average of all cements of a given type.

The aggregate combinations used in the slabs were (1) the sand and crushed stone used in the Missouri test road, (2) the sand and gravel used in the New York test road, and (3) a New York State sand of doubtful quality combined with gravel from the Chicago area. Aggregate combinations used in the boxes and columns were (1) the local Chicago area sand and gravel and (2) the New York State sand of doubtful quality in combination with the Chicago gravel.

Almost without exception the flat slabs and columns are in excellent condition after 8 years' exposure. One or two of the slabs show minor D-cracks, and some pattern cracking has developed on the columns. On the other hand, a number of the boxes, particularly those made with the lean, wet mixes, are showing marked deterioration, some of them to the point of complete disintegration. The boxes have been kept filled with sand saturated with water almost continuously since construction and this fact no doubt accounts for the relatively faster rate of deterioration. It also accounts for the cracking which has occurred in many of the boxes. In such cases, efforts were made to preserve the integrity of the boxes by means of wooden yokes and by sealing the cracks. This maintenance operation was reasonably successful.

As in the case of the other projects, a complete report of progress on the Naperville Farm is being prepared for publication. This report will discuss the whole project in detail, including construction features, maintenance operations and the construction and behavior of numerous supplementary specimens. In the present paper it is proposed to discuss only the box specimens containing the type I and type II cements and the air-entraining counterparts of five of these cements—18 cements in all. All of the specimens have, of course, been rated periodically as to condition by representatives of the Long-Time Study Committee, and this information will appear in the progress report.

The data shown in Table IV are taken from the record of the last official inspection made in May, 1949. A word as to the method of rating. The four sides and the top of each box were rated separately and assigned a numerical value ranging from 1 to 6 depending upon condition, 1 representing no change from the original condition and 6 representing complete disintegration. The very first sign of possible trouble is indicated by 1+. The final rating for the box as given in the table was then calculated by obtaining the numerical average of the rating of the various elements. This accounts for the decimal fractions.

TABLE IV.—PROJECT 10A. NAPERVILLE FARM. RATING[a] OF BOX SPECIMENS WITH RESPECT TO RELATIVE DETERIORATION DUE TO WEATHERING.

Ratings as of May 1949—Specimens cast fall of 1941.

Cement	Cement Content—6 sacks per cu. yd.						Cement Content—4½ sacks per cu. yd.					
	Good Sand			Doubtful Sand			Good Sand			Doubtful Sand		
	1½-in. Slump	3-in. Slump	8-in. Slump	1½-in. Slump	3-in. Slump	8-in. Slump	1½-in. Slump	3-in. Slump	8-in. Slump	1½-in. Slump	3-in. Slump	8-in. Slump
	1	*2*	*3*	*4*	*5*	*6*	*7*	*8*	*9*	*10*	*11*	*12*
No. 11	1	1	1	1+	1	1.5	1+	1	1.8	1+	1.8	5.2
No. 12	1+	1	1	1	1	2.2	1	1	1.3	1	1	3.2
No. 13	1	1+	1	1	1.1	1.2	1	1	1.6	1.1	1.1	5.9
No. 14	1	1	1+	1+	1+	4.8	1.1	1+	1.2	1.8	1+	1.9
No. 15	1	1	1+	1	1	4.0	1	1	1.3	1+	1+	5.9
No. 16	1+	1	1.4	1.1	1	4.6	1	1	1.3	1+	1+	2.3
No. 17	1+	1	1	1	1+	1.1	1	1	1.1	1+	1+	4.8
No. 18	1+	1	1	1	1+	3.0	1	1	2.7	1	1+	4.7
Avg. Type I	1	1	1+	1+	1+	2.8	1	1	1.5	1.1	1.1	4.2
No. 21	1.6	1	1	4.9	1	1.6	1	1	1+	1	1+	1+
No. 22	1.1	1	1+	1	1+	5.0	1+	1	1.2	1.1	1+	3.8
No. 23	1.1	1+	1+	1.4	1	2.7	1	1.1	2.6	1.1	1+	4.8
No. 24	1+	1	1	1+	1+	1+	1.2	1	1.4	1.1	1	4.4
No. 25	1	1	1	1+	1+	1	1	1	1.1	1	1+	1.8
Avg. Type II	1.2	1	1	1.8	1+	2.3	1+	1+	1.5	1.1	1+	3.2
No. 11 T	1+	1+	1	1+	1	1	1+	1	1+	1+	1	1
No. 12 T	1	1+	1	1	1+	1	1	1	1	1+	1	1
No. 16 T	1	1	1	1	1	1+	1+	1+	1	1	1	1
No. 18 T	1	1	1	1	1+	1	1+	1	1	1	1	1
No 21 T	1	1	1+	1	1	1	1	1+	1	1	1	1
Avg	1	1	1	1	1	1	1+	1	1	1	1	1

[a] Rating 1 indicates perfect box. No change from original rating. Rating 6 indicates complete disintegration. Intermediate values denote relative stages of deterioration. The ratings shown above are the numerical averages of ratings for the sides and tops of the boxes. In general, greater deterioration was noted on the south and east faces than on the north and west faces of the boxes.

Let us discuss Table IV. In the first place, it will be observed that the 54 boxes containing the 6-sack mix with the good sand (first three columns) are with two exceptions still all in good condition. This applies to all cements and to all three consistencies, with a slight edge in favor of the 3-in. slump concrete. With the doubtful sand (columns 4 to 6) it is another story. Here most of the boxes containing the 8-in. slump concrete are well on the way to complete failure, whereas the boxes containing the 1½- and 3-in. slump concrete are, with two exceptions, still good. Here again the edge is somewhat in favor of the 3-in. hand-placed concrete as compared with the 1½-in. slump vibrated concrete. Turning now to the 4½-sack mix and noting first the boxes containing the good sand (columns 7 to 9), we find considerably more deterioration in the 8-in. slump concrete than was found in the case of the corresponding 6-sack mix. Ten out of 13 boxes show ratings in excess of 1.1 as compared with only 1 out of 13 for the 6-sack mix. The next comparison is the 4½-sack mix with the doubtful sand (columns 9 to 12). Here again we find the greatest deterioration in the 8-in. slump concrete, the ratings being substantially higher than for the corresponding 6-sack mix with the same sand. Most of these trends would be expected and are in the direction indicated by our prior knowledge of and experience with concrete.

Let us now turn to a comparison of cement types. So far as the $1\frac{1}{2}$- and 3-in. slump concretes are concerned, there is practically no choice. The average ratings show a slight advantage in favor of type I, but this is due to the rather high ratings assigned to certain boxes containing one of the type II cements and probably should be discounted. In the case of the 8-in. slump concrete, there is a slight advantage in favor of type II for both mixes containing the doubtful sand. However, the cements individually vary over a wide range. Moreover, the individual variations are not consistent. For example, the box containing cement No. 14 in the $4\frac{1}{2}$-sack mix with the doubtful sand, 8-in. slump (column 12) has a rating of 1.9 as compared to a rating of 4.8 for the same combination in the 6-sack mix (column 6), a relationship which is distinctly out of line. Note also that cement 21 in the 6-sack mix with the doubtful sand, $1\frac{1}{2}$-in. slump (column 4) has a rating of 4.9 which is far out of line with other boxes containing this cement. Other individual inconsistencies may be noted. These departures from the general trend indicate that the quality of the concrete in these particular boxes was probably influenced more by accidental variations in some construction procedure, such as placing or curing, than by the materials used in the mix.

As in the case of both the New York test road and the Florence Lake Dam, the really outstanding comparison at Naperville so far is the performance of the boxes containing the air-entrained concrete. None of these boxes (60 in all) show a rating in excess of 1+, which means virtually no deterioration at all. This even applies to the lean wet mixtures (high water-cement ratios) and to the concrete containing the doubtful sand. This fact calls for a word of caution. As the result of several years' field experience, the value of air entrainment to improve durability has become well established. However, it has also been shown that air entrainment cannot be depended upon to correct deficiencies due to poor quality of either the aggregates or the concrete. In other words, the fundamental rules governing the making of good concrete apply just as much when air is entrained as when it is not. The only difference is that air entrainment gives us additional protection which even good concrete may not give without it. This truth will probably be demonstrated in time at Naperville.

SUMMARY

Up to the present time the indications of the Long-Time Study are definitely that there is no choice between type I and type II cement in so far as durability or resistance to natural weathering is concerned. This observation is based on general trends in the behavior of concrete containing eight type I cements and five type II cements in four of the Long-Time Study projects, all of which are located in areas subject to severe frost action. So far as the New York test road is concerned, it is believed that, with the possible exception of one cement, this trend will be maintained in the future and that the ultimate life of this pavement will probably be governed more by its ability to resist structural breakdown at joints and cracks than by differences in weathering characteristics due to the use of different cements. This prediction is based on many years' experience in observing the behavior of concrete pavements in service. In general, defects such as D-cracks, if they occur at all, will usually develop within the first 6 or 7 years of the life of the pavement. If they do not occur within this period the chances are that the concrete will be durable and that ultimate failure, if and when it takes place, will be due to other causes.

The relatively poor record of cement 21 on the test road, and at Florence Lake, has not been explained. This cement passed all of the prescribed tests and would be accepted under most specifications. Its behavior at Naperville was erratic, the boxes made with the 4½-sack mix being generally in better condition than the boxes containing the 6-sack mix. A further study of the records may reveal the reason for the relatively poor showing of this cement. However, it should again be noted that cement 21T, the air-entraining cement from the same clinker, has proved entirely satisfactory up to the present time.

The above comments regarding the performance of the New York test road do not mean that the quality of the cement is not important. It is important. However, in my judgment, merely specifying the present A.S.T.M. type II cement will not in itself insure more weather-resistant concrete than would the use of type I. Further research into the question of cement quality as affecting durability is needed. It is unfortunate that the Long-Time Study did not include some cements of the type manufactured 25 years or so ago. A project along this line, involving however the product of one cement mill only, is being started in Kansas through the construction of an experimental road. Sections of pavement will be built with cement from this mill as manufactured today and also as manufactured in 1924. A third section with cement of modern composition but coarse ground will also be laid. This experiment will be followed with interest.

It is recognized that the above conclusion regarding type II cement runs counter to certain theoretical considerations and, laboratory test data to which reference has been made and which do indicate that a reduction in the C_3A compound is desirable. It is also recognized that so far as the Long-Time Study is concerned, the final answer will probably not be forthcoming for several years. However, it should be pointed out that, in addition to the present indications of the study, we have the results of many years' experience with high C_3A cements in structures which have given excellent service. A classic example is the old Ohio Post Road built in 1914–15. This pavement, located on U. S. 40 between Zanesville and Columbus, Ohio, proved exceptionally durable in service and was replaced finally after many years only because of obsolescence. The cement used in this pavement was high in C_3A. It would now be classified as a typical type I cement except that it would not have conformed to the present A.S.T.M. requirements for fineness. This example, as well as others, indicate that merely specifying type II cement is not the answer to the problem of durability. The answer lies much deeper and the only way to find it is through continued research into the properties of cement as they are affected by composition and by methods of manufacture.

DISCUSSION

MR. BAILEY TREMPER[1] (*presented in written form*).—The author has considered the effect of type of cement on the durability of concrete mainly from the standpoint of resistance to freezing and thawing. If resistance to this type of exposure is needed, the appropriate measure appears to be the use of air entrainment, and this has been pointed out clearly by the author. In such concrete, possible differences between type I and type II cements in their resistance to freezing and thawing alone may be considered to be of minor significance.

In the writer's opinion, the case for type II cement lies, not in superior resistance to severe weather exposure, but in greater constancy of volume, a factor that is touched upon but lightly in the author's discussion. The writer refers particularly to the property of drying shrinkage.

Investigations by Carlson[2] and Gonnerman[3] demonstrate that, other factors being equal, cements with the higher C_3A content shrink more in drying. A similar conclusion may be reached by analysis of the data presented by Lerch[4] in his study of the effect of gypsum in cement.

Since type I cement may have a relatively high content of C_3A and type II not over 8 per cent, the differences in drying shrinkage may be considerable. High drying shrinkage may be a factor in poor durability under conditions not related to freezing and thawing, as the writer will attempt to demonstrate in the succeeding paragraphs.

The accompanying Fig. 1 is a view of an area of one side of a heavily-traveled four-lane highway in the State of Washington in a location where winters normally are mild. The view shown is typical of areas in the poorest condition over a distance of 15 miles. The major portion of the balance of the section is affected similarly, but to a degree which, while readily visible on inspection, is difficult to portray adequately in printed form. The photo was taken 18 yr. after construction of the pavement, just before it was necessary to apply a resurface. The closely spaced cracks in random pattern appear to be so deep as to have caused severe structural damage to the slabs. The concrete in this pavement has been observed with interest over a period of years. It seems evident that the majority of the present deepseated cracks have formed at the locations of what were originally shallow surface cracks. The latter apparently were caused by severe drying shrinkage at the cessation of wet curing. The present cracks are most prominent at slab ends, indicating that severe flexing caused by heavy traffic has deepened the originally shallow shrinkage cracks. The belief that drying shrinkage was a primary cause of the distress is strengthened by the condition of the two-lane pavement on the opposite side of the highway which does not show this type of cracking. It was built two years earlier to the same design and with the same aggregates. The cement, however, was from a different mill. The latter contained 9 per cent tricalcium aluminate while that used in the defective pavement contained 15 per cent.

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[1] Materials and Research Engineer, Department of Highways, Olympia, Wash.

[2] Roy W. Carlson, "Drying Shrinkage as Affected by Many Variables," *Proceedings*, Am. Soc. Testing Mats., Vol. 38, Part II, p. 419 (1938).

[3] H. F. Gonnerman, "Study of Cement Composition in Relation to Strength, Length Changes, Resistance to Sulfate Waters and to Freezing-and-Thawing, of Mortars and Concrete," *Proceedings*, Am. Soc. Testing Mats., Vol. 34, Part II, p. 244 (1934).

[4] William Lerch, "The Influence of Gypsum on the Hydration and Properties of Portland Cement Pastes," *Proceedings*, Am. Soc. Testing Mats., Vol. 46, p. 1252 (1946).

It is not claimed that the difference in tricalcium aluminate alone was responsible for the difference in performance although it is believed to have been a contributing factor. Differences in alkalies and possibly in fineness of grinding also are believed to have affected the degree of drying shrinkage.

Similar comparisons between performance of cements in pavements have been noted elsewhere in Washington. Traffic, however, has not as yet been heavy enough elsewhere to cause the severe structural damage of the instance cited above.

In the Pacific Northwest, at least, type II cement may be obtained as readily as type I. Differences in manufacturing costs do not appear to be sufficient to affect the selling price. In fact, some manufacturers have stocked but one cement and have marketed it as either type in accordance with demand.

It is the writer's belief that any steps that may be taken through specification requirements to reduce the degree of drying shrinkage are justified, particularly when such protection can be obtained at no extra cost. The use of type II cement with its lower mandatory C_3A content is one of the means of obtaining such protection.

As far as the writer can determine, air-entrainment does not consistently affect the degree of drying shrinkage. If such is the case, maximum protection against severe drying shrinkage must be obtained through specifications for the cement, whether or not the concrete is air-entraining.

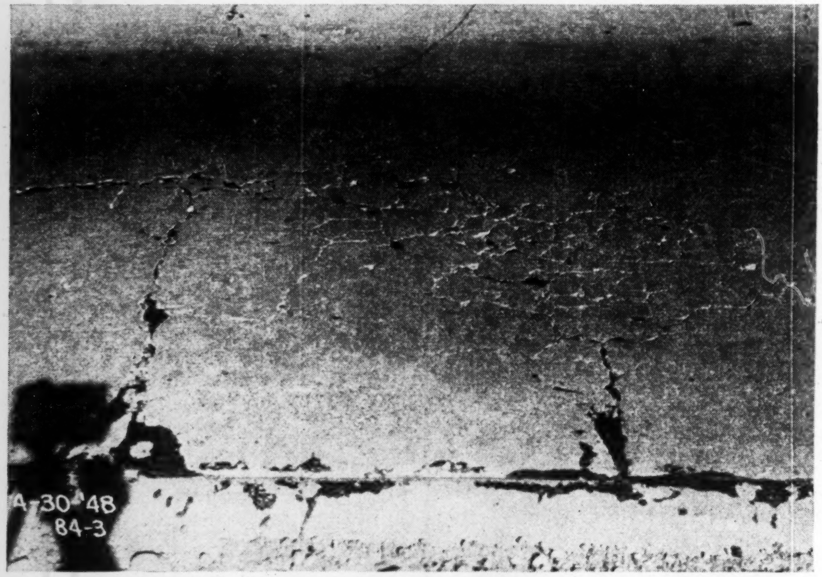

FIG. 1.—One Side of a Heavily-Travelled Four-Lane Highway in the State of Washington.

Lerch[4] has found, and a working subcommittee of A.S.T.M. Committee C-1 on Cement has confirmed, that drying shrinkage may be reduced markedly if the SO_3 content is controlled at its optimum value. When, and if, a requirement for optimum SO_3 becomes a part of portland-cement specifications, the advantage of using type II cement, where heat of hydration and sulfate resistance are not factors, presumably will disappear. Until that time, the writer is firmly convinced that type II cement, plus air-entrainment when needed, gives the greater assurance of durability.

CHAIRMAN T. E. STANTON.[5]—A very pertinent field of investigation would appear to be the study of whether or not differences of C_3A content have any important bearing on the drying shrinkage of the concrete, or whether some other factors are responsible.

I think that we will agree that that is a field that can be explored to considerable advantage.

MR. H. F. GONNERMAN.[6]—It may be of interest to present some field observations of the performance of types I and II cements.

In the early 1930's when the question of cement composition was active, a survey was made of some 659 projects comprising 2386 miles of concrete pavement in New York State where very severe weathering conditions are encountered. Our primary interest in making this survey was to study the surface scaling that began to appear in some of the concrete pavements about that time. Previous to 1930, New York State had not used salts to any great extent to keep pavements free from ice. But in 1931, or thereabouts, the state began to use salts, mixed with sand or cinders, at intersections and on hills and curves; at times the salts were used raw without mixing with cinders or sand. This practice caused scaling of the surface of the pavements at those locations where the salts were applied.

This survey was made during the summer and early fall of 1933. Such information as was available was obtained on the cements used from 1916 to 1928, the period during which the pavements surveyed were constructed. No pavement less than 6 years old was included in the survey. The approximate compound compositions of the cements were computed and the performance of the pavements was then studied from the standpoint of the computed C_3S and C_3A contents of the cements. A wide variety of aggregates was used in the pavements, some good and some of doubtful quality. Although these and other uncertainties were in the picture, we tried to determine if there were any trends in the performance of the pavements that could be attributed to the composition of the cements. Such trends as were found in the over-all performance of the various cements with respect to C_3S and C_3A content appeared to be in favor of the cements having the higher C_3S contents and the lower C_3A contents. Later we discovered that these results were clouded by the fact that some of the cements that showed good performance in this survey contained crusher oil, which unintentionally got into the cement from the mills during grinding of the cement. The crusher oil produced some air entrainment in the concrete which greatly improved its resistance to surface scaling and to other forms of deterioration caused by freezing and thawing.

In order to obtain more accurate information regarding the effect of the composition of the cement on pavement performance, during the period 1935 to 1938, New York State constructed thirteen experimental concrete highway projects in which special commercial cements of varying chemical composition were employed. These special cements included:

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[5] Materials and Research Engineer, Division of Highways, Sacramento, Calif.

[6] Assistant to Vice-President in Charge of Research and Development, Portland Cement Assn., Chicago, Ill.

1. Cements low in C_3A and both low and high in C_3S (in the range of A.S.T.M. type II).

2. Cements high in C_3A and both low and high in C_3S content (in the range of A.S.T.M. type I).

3. Cements both low and high in C_3A but high in C_3S content (in the range of A.S.T.M. type III).

Blends consisting of 5 or 6 bags of these various portland cements mixed with 1 or 2 bags of two brands of natural cement were also used in the experimental projects.

It was found that the composition of the cement could not be correlated with the performance of the pavements either as regards surface scaling or appearance of so-called D-cracks. However, these projects did bring out clearly that the portland cements which inadvertently contained crusher oil gave outstanding performance as did many of the blends of portland and natural cement where the latter contained fat added during grinding of the cement.

Then, in cooperation with the New York State Highway Department and with other state highway departments, other experimental roads were built during 1938 to 1942 with cements in which some of the air-entraining agents which we had experimented with in the laboratory were purposely interground with the cement. We have followed the performance of these roads since their construction and to date have found no surface scaling on any of them regardless of the type of cement used, in which the concrete had the proper amount of air entrainment.

It may be of interest to point out that in February, 1934, the Portland Cement Assn. submitted to A.S.T.M. Committee C-1 proposed tentative specifications covering two types of portland cement, (types IV and V) in addition to the two types (types I and III) then in current use. These proposed specifications were not accepted by Committee C-1 at that time. In 1938, or thereabouts, they were brought up again by a special committee of users on Committee C-1 of which Mr. Jackson was chairman. This special committee recommended specifications for five types of portland cement as pointed out by Mr. Jackson in his paper. Upon the adoption in 1940 of these tentative specifications covering five types of portland cement, four brands each of the five types specified were purchased in the open market and subjected to various tests in the laboratory, among which was a freezing-and-thawing test. In order to give each type of cement an even break the specimens were moist cured different lengths of time so that when subjected to freezing and thawing they would all be of approximately equal strength. We found among the type II cements some that did not perform as well as some of the type I cements and *vice versa*. This is in agreement with the results presented by Mr. Jackson.

Recently we had occasion to review data presented in the First and Second Interim Reports on Concrete Research of the Corps of Engineers, U. S. Army, on some fifty cements, which were subjected to freezing and thawing in sea water at Eastport, Maine, where during one winter 150 or more cycles of freezing and thawing are obtained. Upon dividing these cements into type I and type II cements on the basis of their chemical analyses, we found that among the non-air-entraining cements of lowest resistance to freezing and thawing were a number of type II cements and some type I cements and among the non-air-entraining cements of highest resistance were some type I cements as well as some type II cements. Regardless of whether they were type I or type II, the six cements containing air-entraining agents gave by far the best performance in these tests.

Laboratory studies are being made to determine to what extent the composition of the cement affects the durability of concrete. In one series now nearing completion, concretes made from non-air-entraining cements of varying composition and fineness were subjected to freezing and thawing after various periods of curing. It so happens that in this group of cements is one of type II composition which, when subjected to freezing and thawing after curing 28 days in the moist room, exhibited very low resistance to freezing and thawing. However, when the air content of concrete made with this cement was increased from about 0.7 per cent to 1.5 per cent by adding Vinsol resin in solution during mixing, the resistance of the concrete was increased from 5 cycles to about 220 cycles. With 2.6 per cent air the resistance was increased to 700 or more cycles and with 4.6 per cent air the specimens have already withstood 1400 cycles and are still under test. Thus with air entrainment the resistance of the concrete to freezing and thawing was tremendously increased over that obtained with concrete without air entrainment. So far as frost resistance is concerned, this work confirms the conclusions drawn by Mr. Jackson in his paper regarding the marked benefits of air entrainment regardless of the type or composition of the cement.

Other factors are being studied in an effort to determine their effect on the frost resistance of concrete. Eventually it is hoped to isolate those factors that make for good durability, and thus be able to design the kind of concrete best suited for pavements and structures which are to be exposed to severe weathering.

MR. W. C. HANNA.[7]—I would like to point out that some cements on the market meet all the specification requirements for type I and all the specification requirements for type II cement. Therefore, there can be no difference between type I and type II, and that is a very satisfactory way of ironing out these arguments.

MR. M. A. SWAYZE.[8]—I feel that we should say something about a survey that our research laboratory made some ten to eleven years ago. It took us two years to examine every one of the 45 individual cements made by our company. These were type I, type II, and type III cements. I will confine my remarks largely to the difference in the type I's and II's, of which there were a goodly number.

We made concrete tests on a wide range of cement contents with two consistencies. We ran strength tests, volume change tests and tests for resistance to freezing and thawing. In these tests there was no doubt when we got through that the type I cements in general—and I am now talking of the real type I, that is where the C_3A content was above the 8 per cent limit and not what has been spoken of before where they happened to meet the requirements of both specifications—had definitely higher resistance to freezing and thawing than did the type II products.

There were exceptions and those are worthy of note. There was one type II cement that was outstanding in resistance and that is the same one that Mr. Gonnerman mentioned in New York. It had a slight addition of crusher oil which we couldn't keep out. That cement had the best durability in freezing of any in the list of 45.

Omitting this product, it was very

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[7] Chief Chemist, California Portland Cement Co., Colton, Calif.

[8] Director of Research, Lone Star Cement Corp., New York, N. Y.

definite that the general run of type I products were better than type II so far as frost resistance was concerned.

There were other exceptions, and these again are worthy of note. There are some high-silica portland cements where the silica ratio is exceedingly high, and in every case these cements had lower durability. Tracing their history, it came down to a lack of burning, whether or not the composition otherwise was type I or type II. Wherever you run into a cement that is very hard to burn, you are running into danger of low durability to frost.

We think it is far more important not to look so much to cement composition but to what we do with the concrete after we get it on the job—how we mix that concrete and how we lay it on the grade, the time we spend in finishing and the time we spend in curing. Once we concentrate our attention on the mode of use of cement rather than on slight variations in composition, I believe we will get more durable concrete.

MR. C. H. SCHOLER.[9]—In studying the problem of shrinkage, particularly, we want to remember that we get from 100 to 300 per cent more variation in shrinkage with the aggregates than we do with the cement; and that is an important element in the study of concrete. The aggregates are of extreme importance with regard to shrinkage.

I have examined about 9000 miles of concrete pavement with about 90 different cements. Those who read the report on this investigation will find the kind of concrete you make is more important than the type of cement used.

I think that if you made an alkali resistant concrete out of an extremely high tricalcium aluminate cement you would be making good enough concrete.

CHAIRMAN STANTON.—Comments from the Chair may not be in order, entirely, but I agree absolutely with what Mr. Scholer has said: "If you make good concrete"—but we don't always make good concrete. Therefore I think the insurance we may get under certain conditions, such as alkali or low temperature exposure, justify the use of special types of cement in structures which may be subjected to such severe exposure conditions.

MR. R. E. DAVIS.[10]—I wish to ask a question. Just a moment ago we heard that cements that were high in silica were generally less resistant to freezing and thawing. I would like to ask at what age those tests were started. It is generally recognized that some cements are slower hardening than others, and I think that the results of freezing-and-thawing tests in the laboratory will greatly depend upon the age at which the tests are started.

When comparing cements, the results of tests started at 28 days may lead to conclusions which are quite different from those based on tests started at 6 months.

MR. SCHOLER.—Those cements were not low-heat or otherwise low-strength cements. They were either high-strength cement or cements passing type I or type II requirements so far as composition is concerned. They had perfectly normal strength except that it was our observation that the strength gained was somewhat plastic.

In all cases the freezing-and-thawing tests were made by curing the cement one day in the mold, 27 days in fog at 70 F., and then the cycles of freezing and thawing were started.

MR. DAVIS.—That is my criticism of a good many freezing-and-thawing tests. We overlook the fact that in the laboratory concrete at the age of 28 days is subject to much more severe freezing and thawing conditions than are normally found in nature.

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[9] Professor of Applied Mechanics, Kansas State College of Agriculture and Applied Science, Road Materials Lab., Manhattan, Kans.

[10] Professor of Civil Engineering, and Director, Engineering Materials Lab., University of California, Berkeley, Calif.

Among type I and type II cements there are wide variations in early-age strength characteristics. Regardless of their ultimate strength in concrete, those which exhibit relatively high early strength will show superior performance in tests started at 28 days. Those exhibiting high later age strength will show superior performance in tests started at, say, 3 months.

We will find corresponding performance variations among cements in field structures. A "fast" cement will show up well in concrete placed in late fall or winter; a "slow" cement will show up poorly unless the concrete is given adequate early-age protection against freezing. But for the same two cements, the reverse may be true when the concrete is placed during the summer.

In judging durability as affected by freezing and thawing, it therefore seems clear that early environmental conditions of the structure ought to be taken into consideration. For average field conditions, probably the results of accelerated laboratory tests started at the age of four or five months are much more indicative of what may happen in nature than are the results of accelerated tests started at the tender age of 28 days.

MR. N. T. STADTFELD.[11]—I think the one thing that ought to be stressed a little more is the possibility that type II cement may be more uniform. And I say that quite seriously because in the old days when we used to get type I cement the alumina content might differ from $4\frac{1}{2}$ per cent to $7\frac{1}{2}$ per cent from week to week. We would build a fence and have three panels perfect. The fourth might be made under exactly the same conditions but would deteriorate before the contractor left the job.

This is what caused Mr. Merriman to start his investigations. If any of you, who are from Los Angeles, will take a look at the Los Angeles River and the slopes thereof which have been paved with concrete using Merriman cement, you will see that it is one of the monuments to his name.

We have had the same experiences in New York City where we have used this cement, now, for 12 years.

But referring to the Jackson paper—outside of uniformity, it seems to me that in type II cement, which it was hoped would perform in a certain way and which in long-time studies may not have showed up as well as we had hoped, the trouble has been in the way in which the specifications were written for type II cement.

For years, I have tried to raise the standard of this cement by requiring a higher strength for the 7-day mortar test. I am prepared to say that if any of you get type II cement which hovers around 250 psi. for the 7-day mortar briquet, you had better be a little suspicious as to how well that cement has been burned, because in our case where we have control and know how well the cement is burned, we have never fallen below 300 psi. and consider 350 psi. a fair average. Now, it seems to me that the next step is an improvement in the requirements of that specification.

Mr. Jackson has stated only one point, and that is the resistance to freezing and thawing. Mr. Swayze has taken up the 24 per cent silica or high-silica cement. It is perfectly true that in our experiments, even in hard burning, we have to watch the high-silica cement in order to get early strength. This is nothing against the high-silica cement, except that a contractor wants to remove his forms within a reasonable time. With our particular cement, we can allow him to move an arch form after 6 hr. The usual time is 8 hr.

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[11] Division Engineer, Inspection and Testing Div., Board of Water Supply, New York, N. Y.

I want to emphasize that simply a C_3A requirement means very, very little. We have to go much farther than composition.

The following will make this clear. When Mr. Merriman wrote the specifications for the Fort Peck spillway and retaining walls he was told by a representative of the industry that all that was required was to have the specifications call for a maximum C_3A of 7 per cent.

I want to emphasize here the fallacy of any such contention. We had to have a fairly high resistant cement against sulfates, and the specifications were written accordingly. That was the first sulfate-resistant cement that I knew of in the country.

But, from one mill, with composition exactly the same from day to day due to the narrow limits of the chemical requirements, the sulfate resistance test which Mr. Merriman started—and which, to my mind, is still a very good one—would pass at certain times and at other times the cement would simply not pass that test. When we investigated what the difference might be, it turned out that some new coal burning apparatus had been installed and we were not getting the proper heat treatment in the kiln.

I still believe that we are on the right track when we say that manufacturing control is of the greatest importance. You will find differences in cements, possibly not only between type I and type II, but among the various cements of type I and among the various cements of type II, depending on what goes on in the kiln room.

MR. R. F. BLANKS.[12]—I am very sorry that Mr. Jackson has seen fit to make the presentation that he has at the present time. I consider it premature and ill-advised.

The reason I say that is this. Mr. Jackson has asked a question, and then he doesn't answer it. Whenever you ask a question in the technical literature and do not follow with a satisfactory answer, it leads to confusion and nothing else.

If we take the question as he has it, "Why Type II Cement?," I am afraid he has not even come close to answering the question. We know from the record that type II cement has certain qualities which are far superior to those of type I cement.

If we take the question as discussed in the paper, "Does Type II Cement Make Concrete More Resistant to Severe Freezing and Thawing" we could have told him a long time ago that the answer to that was, "no." In other words, we don't make concrete highly resistant to freezing and thawing by using type II cement.

He should have asked the question "Why Air-Entrained Concrete?" To that question, he has given a complete answer.

MR. F. H. JACKSON (*author's closure*).—Mr. Tremper indicates that, in his opinion, the case for type II cement lies not so much in its superior resistance to freezing and thawing as in its greater constancy of volume. Granted that many laboratory investigations, including those cited by Mr. Tremper, do indicate a relationship between calculated C_3A and drying shrinkage, the author would like to point out that, up to the present time, none of the projects of the "Long-Time Study of Cement Performance in Concrete" indicate that differences in C_3A ranging from 3.7 to 13.2 per cent (the range covered by the 8 type I and the 5 type II cements represented in the study) has had any observable effect upon the early cracking tendency of the concrete. Furthermore, some of these projects were built under conditions which it might be assumed would tend to bring out differences of this nature. For example, the Missouri test road was built during a period of hot, relatively dry weather. On this project measured temperatures of concrete at time of placing never fell below 80 F. and averaged around 90 F. Construction was started on July 31, 1941, and completed on August 8, 1941 and up to the time of the last official inspection there were no indications whatever that such minor differences in behavior as had developed could in any way be related to differences in the volume change characteristics of the individual cements. These facts would indicate to the author that variations in computed C_3A within the range represented in the long-time study, while they may possibly be of interest from a theoretical standpoint, are not of practical importance as affecting the relative amount of initial cracking that may take place in the concrete structures in which they are used.

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[12] Chief, Research and Geology Engineering Control and Research, U. S. Bureau of Reclamation, Denver, Colo.

The author believes that there may be a plausible explanation for the lack of correlation between the results of laboratory tests for drying shrinkage and field performance. Laboratory specimens are almost always stored under conditions which minimize restraint and all surfaces are exposed to drying. The usual field member is always restrained and seldom are all the surfaces exposed. Furthermore, concrete in the field is seldom exposed to continuous drying as is the laboratory specimen. Also, under field conditions, moisture and temperature effects usually tend to offset each other, drying out being frequently accompanied by a rise in temperature and *vice versa*. All of these variables would tend to reduce the observed effect of the comparatively small differences in drying shrinkage which might result from variations in the composition of the cement.

In reply to Mr. Blanks, the author would like to point out that, although the title of the paper is perhaps too broad, the exact scope of the discussion is clearly stated in both the Synopsis and in the opening paragraph of the paper itself. Perhaps a more descriptive but certainly too long a title would be "Can Type II Cement be Justified as an Improved General Purpose Cement, Particularly from the Standpoint of Improved Resistance to Freezing and Thawing?" In the author's opinion, the paper supplies a rather convincing answer to this question—and that answer is "No." Of course the extent to which the reader is willing to accept this conclusion will depend upon his willingness to accept the evidence of eight years exposure in the long-time study tests. Mr. Blanks, judging from his comment, appears to agree with the answer, at least in so far as it applies to resistance to severe freezing and thawing. However, as brought out in the paper, there are many engineers, particularly in the eastern part of the country, who continue to specify type II cement (or type IIA if they want air entrainment), apparently with the idea that there is some magic in the type II composition which will *per se* give them improved durability. The author is firmly convinced that the comparatively small differences in the composition in a typical high C_3A type I cement as compared to a typical type II cement are not sufficient to have any practical effect upon durability. He agrees with Mr. Swayze and others who feel that the durability of concrete is affected to a far greater extent by the care used in proportioning, mixing, placing, and curing than by small variations in the chemical composition of the cement used in the mix.

As Mr. Blanks pointed out, the best way to secure frost-resistant concrete is by the use of air entrainment. This has been clearly demonstrated by the long-time study and other researches as well as by practical experience in the use of air-entrained concrete in the field for a period now extending over ten years. For this type of concrete, the question of type II *versus* type I cement would seem to be even less important than it has been shown to be in the case of non-air-entrained concrete. This also has been demonstrated by the long-time study tests. For example, in the Naperville project, all the concrete specimens containing the air-entraining cements are still in excellent condition after eight years' exposure to severe weathering. This is true of all six of the air-entraining cements regardless of type. The same may be said for the other three projects referred to in the paper.

It should not be assumed from the above comments that the author considers the quality of the cement to be unimportant. It is very important and it may very well be that, as the result of continuous and intensive research in this field, important changes in both composition and fineness will be indicated. The author simply does not agree that any significant improvement in durability is effected by merely specifying type II cement.

Along the line of Mr. Hanna's comment, it is quite true that some cements will meet all of the specification requirements of type I and type II. Practically all type II cements will meet the type I requirements. On the other hand, he should know that there are many type I cements that will not meet the requirements for type II. Of the thirteen cements in the long-time study that were classified as type I or type II, eight did not meet the requirements for type II. They were either too high in C_3A or C_2S or both. Mr. Hanna of course knows that it is the usual practice in discussions of this subject to consider as type I only those cements which cannot be classified as type II and this practice was followed in the paper.

LONG-TIME STUDY OF CEMENT PERFORMANCE IN CONCRETE
CHAPTER 6—THE HEATS OF HYDRATION OF THE CEMENTS[1]

By George J. Verbeck[2] and Cecil W. Foster[2]

Foreword

This paper is the sixth in a series describing and presenting results from the research project titled, "The Long-Time Study of Cement Performance in Concrete." This project, which was undertaken by the Portland Cement Association in 1940, was the direct outgrowth of a suggestion made by P. H. Bates, Past President, A.S.T.M., then Chief, Clay and Silicate Products Division, National Bureau of Standards. The plan of the investigation, as contemplated by Mr. Bates, comprised two essential features, both of which were incorporated in the test program.

The first of these features was the use of a representative group of American portland cements in building several series of concrete structures, or portions of structures, which would be observed over the years to learn the extent to which differences in cement characteristics influence the durability of concrete. The series of structures were to be built so that the different cements would be used under identical conditions in each of several locations selected to provide a considerable range in exposure conditions.

The second essential feature of this program was the inauguration of laboratory studies designed to explain, in terms of composition or other properties of the cements, any differences in durability which may develop from the exposure studies. To this end it was arranged to make all of the known or accepted tests of cement and such other tests as seemed to offer promise, and to preserve an ample supply of the cements for any new tests which may be developed or for additional field investigations that are suggested as the study unfolds. This paper is a report on certain studies dealing with heat of hydration.

Since this is the first report of this investigation to be printed by the A.S.T.M., a brief résumé of the program is presented.

The program was developed by an Advisory Committee, under the Chairmanship of P. H. Bates, consisting of leading authorities in the fields of cement and concrete and representatives from the Portland Cement Association and its member companies. In addition to developing the program and selecting the cements to be used, this Committee has followed the investigation closely and reviewed all reports and published papers.

The cements selected were ground from 21 clinkers produced at 14 different plants representing geographically the principal cement-producing centers of the United States. All five A.S.T.M. types were included. Six of the 21 clinkers were ground with flake Vinsol resin to produce six air-entraining cements, giving a total of 27 test cements. A total of 37,000 bbl. was produced for the investigation. Sixty barrels of each are preserved for future requirements. When the cements were being manufactured, observers from the Research Laboratory of the Portland Cement Association were present collecting samples and recording special features of the operation.

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[1] This paper was presented at the First Pacific Area National Meeting of the Society held in San Francisco, Calif., October 10–14, 1949.

[2] Research Chemists, Applied Research Section, Portland Cement Assn., Chicago, Ill.

The structures for the exposure studies include six miles of highway pavements (in three states); 222 reinforced concrete piles in fresh and salt water (two northern and two southern locations); 180 sections of parapet walls and walkway slabs on dams in high altitudes; 2000 boxes, columns and slabs on exposure plots in Illinois and Georgia; and 1000 test beams in sulfate soils in California. Construction on the projects started in June, 1941, and was completed June, 1943; records of 6 to 8 years' exposure are now available.

Four papers on the project appeared in the *Journal* of the American Concrete Institute, February to June, 1948, as follows: History and Scope of the Project; Manufacture of the Test Cements; Chemical and Physical Tests of the Cements; and Microscopical Study of the Clinker. These were reprinted as *Bulletin 26* of the Research Laboratory of the Portland Cement Association. The fifth chapter of the report on this project entitled "Concrete Exposed to Sulfate Soils," was published as a special publication of the American Concrete Institute and the Portland Cement Association.

When water is added to portland cement, heat is liberated, the rate and amount being a function of the fineness and composition of the cement, the water-cement ratio, and temperature. In concrete, the resultant temperature changes are accompanied by expansion of the concrete while the temperature is rising and by contraction on subsequent cooling. Under most conditions of concrete construction, the heat is dissipated rapidly, and it is only the heat liberated at early ages that is of consequence. In mass concrete where the heat is not readily dissipated, the temperature of the concrete may continue to rise for a considerable time as the cement continues to hydrate. Studies prior to the construction of Hoover Dam stimulated research on the effect of cement composition and fineness on the heat evolved during hydration and hardening. The results of these studies (**1, 2, 3, 4, 5**)[3] led to the development of specifications for low-heat, type IV, portland cements.

The results published in previous papers were confined to tests at ages up to one year or less. The absence of data on the heat of hydration beyond one year has led to considerable speculation as to the quantity of heat liberated at later ages. Based on increases in strength shown at later ages, it might be anticipated that the type IV cements would liberate more heat at later ages than type I cements. The cements used in the Long-Time Study of Cement Performance in Concrete provided an excellent opportunity to obtain information on the heats of hydration of the various types of portland cement. The cements in this series include 21 non-air-entraining cements and 6 air-entraining cements, ground from clinker produced at 14 different plants. The non-air-entraining cements represent one or more of each of the five types described in A.S.T.M. Standard Specifications for Portland Cement, (C 150 - 47).[4] Complete data on the manufacture and properties of these cements have been given in previous chapters of this report. Data on the heats of hydration of these 27 different cements are now available for ages of test up to 6½ yr. These data are presented in this report.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1257.

[4] 1947 Supplement to Book of A.S.T.M. Standards, Part II p. 1; Tentative Revision, 1948 Supplement to Book of A.S.T.M. Standards, Part II, p. 265.

Composition and Fineness of the Cements

The calculated potential compound compositions, minor oxide contents, finenesses and ignition losses of the 27 cements used in this study are shown in Table 6-1-A.[5] These results are from Tables 3-4 and 3-5 of Chapter 3 by Lerch and Ford (6) with the exception of the SO_3 results which are from the original analytical data carried to the second decimal place and the ignition losses which are those of the cements at the time the heat of hydration tests were started. The cements in this table represent eight type I, four type IA, five type II, one type IIA, three type III, one type IIIA, four type IV, and one type V cement. The air-entraining

TABLE 6-1-A.—CALCULATED COMPOUND COMPOSITION, MINOR OXIDE CONTENT AND FINENESS OF CEMENTS.

Cement	Calculated Compounds,[a] per cent by weight				Chemical Analyses, per cent by weight					Specific Surface (A.S.T.M. Method C 115),[c] sq. cm. per g.	Loss on Ignition, per cent by weight
	C_3S	C_2S	C_3A	C_4AF	SO_3	Free CaO	MgO	Na_2O	K_2O		
					Type I and IA Cements						
No. 11	50.0	22.0	12.1	7.2	1.59	0.4	3.7	0.21	0.51	1820	0.86
No. 11T[b]	51.0	21.0	12.2	7.1	1.53	0.5	3.7	0.22	0.51	2010	0.92
No. 12	45.0	28.0	12.6	7.3	1.55	0.1	3.1	0.28	0.40	1815	0.55
No. 12T	46.0	27.0	12.5	7.1	1.54	0.1	3.1	0.31	0.40	1790	0.55
No. 13	50.0	26.0	10.1	6.5	1.62	1.6	1.1	0.04	0.19	1665	1.57
No. 14	42.5	32.0	8.2	9.2	1.73	0.2	2.5	0.06	1.30	1880	0.75
No. 15	64.5	10.0	12.1	7.5	1.92	0.4	0.8	0.08	0.23	1845	0.87
No. 16	53.5	21.0	7.5	10.7	1.71	0.7	2.1	0.23	0.46	1785	0.78
No. 16T	52.5	22.0	7.9	10.4	1.84	0.8	2.0	0.23	0.44	1680	0.78
No. 17	52.0	23.0	10.4	9.3	1.72	0.4	1.1	0.08	0.43	1770	0.61
No. 18	44.5	28.0	13.2	6.8	1.83	0.3	2.6	0.12	0.13	1750	0.92
No. 18T	44.0	28.5	13.2	6.7	1.68	0.4	2.6	0.14	0.13	1960	0.96
					Type II and IIA Cements						
No. 21	40.0	41.0	6.4	9.7	1.24	0.7	1.3	0.22	0.40	1630	0.72
No. 21T	38.0	40.0	6.6	9.7	1.27	0.6	1.3	0.21	0.43	1750	0.75
No. 22	41.5	33.5	6.6	11.7	1.41	0.1	3.2	0.24	0.37	1775	0.51
No. 23	51.0	24.0	3.7	16.6	1.50	0.4	0.9	0.59	0.14	1875	0.46
No. 24	41.0	29.0	5.4	14.8	1.75	0.9	3.1	0.06	1.30	1925	1.30
No. 25	34.0	39.0	4.7	14.9	1.86	0.2	2.2	0.21	0.54	1825	0.37
					Types III and IIIA Cements						
No. 31	56.0	17.0	10.8	6.4	2.23	1.5	3.3	0.23	0.22	2795	1.16
No. 33	60.0	13.0	10.4	7.7	2.31	1.8	1.4	0.21	0.44	2530	1.41
No. 33T	57.0	16.0	10.4	7.3	2.19	1.8	1.5	0.21	0.46	2510	1.31
No. 34	64.0	10.5	5.7	10.1	1.72	2.3	2.5	0.28	0.28	2465	1.24
					Type IV Cements						
No. 41	20.0	51.0	4.5	15.2	1.98	0.4	3.0	0.06	1.19	1915	1.15
No. 42	27.0	55.0	3.5	8.2	1.52	0.2	1.8	0.16	0.26	1920	0.70
No. 43	25.0	48.0	6.2	13.8	2.13	0.1	1.6	1.00	0.08	1965	0.66
No. 43A	29.0	52.0	5.3	9.3	1.87	0.4	1.1	0.33	0.01	1915	0.73
					Type V Cements						
No. 51	41.0	39.0	3.7	10.0	1.40	0.5	1.7	0.08	0.22	2025	0.63

[a] Corrected for free CaO, not corrected for minor oxides. Data are from Tables 3-4 and 3-5 of Chapter 3 (6) with the exception of the SO_3 results which are from the original analytical data carried to the second decimal place, and the losses on ignition which are those of the cements at the time the heat of hydration tests were started.

[b] Letter "T" after cement number indicates cements interground with flake Vinsol resin.

[c] 1949 Book of A.S.T.M. Standards, Part 3, p. 118.

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[5] The numbering followed in this paper for tables and figures differs from that usually used in A.S.T.M. publications. The system followed here is to indicate that this paper is the sixth of a series; therefore the number six is used in each as: Table 6-1 or Fig. 6-1, Table 6-2, etc.

cements are indicated by the letter "T" after the cement number.[6]

The phase compositions of the clinkers from which the cements were prepared were determined microscopically and are shown in Table 6-1-B. These results are from Table 4-2 of Chapter 4 by Brown (**7**) and have been included for use in the least squares analysis of the heat of hydration data in a later section of this report.

TABLE 6-1-B.—MICROSCOPIC COMPOSITION OF K-2 CLINKERS.

This table is a reproduction of Table 4-2, Chapter 4.

Clinker	C_3S	C_2S	C_3A	C_4AF	Dark Prismatic	Glass	Free CaO	MgO	Undifferentiated
TYPE I									
No. 11	57.7	12.8	3.1	2.8	2.3	5.8	0.8	4.0	10.7
No. 12	48.6	22.8	5.6	3.7	3.1	1.6	0.3	2.6	11.7
No. 13	57.8	19.6	1.6	2.2	1.9	8.2	1.2	0.5	7.0
No. 14	52.9	24.3	3.3	8.0	1.0	0.4	0.8	2.5	6.8
No. 15	65.1	6.2	8.6	5.0	1.2	0.3	0.7	0.7	12.2
No. 16	61.4	14.9	4.4	9.2	0.4	1.5	0.9	1.8	5.5
No. 17	51.3	22.6	4.2	4.8	2.7	1.4	0.7	0.8	11.5
No. 18	60.3	16.9	3.8	3.9	2.5	1.6	0.8	0.9	9.3
TYPE II									
No. 21	42.4	37.1	2.0	7.0	0.4	1.7	1.4	1.4	6.8
No. 22	45.6	30.0	2.2	8.2	0.6	0.8	0.2	3.0	9.4
No. 23	55.1	22.4	1.2	6.2	1.5	2.0	0.4	0.4	10.8
No. 24	52.7	20.4	2.9	17.8	0.8	0.0	0.8	3.2	1.4
No. 25	42.2	36.8	1.5	14.6	0.3	0.2	0.2	1.9	2.3
TYPE III									
No. 31	62.1	7.1	3.2	1.9	4.4	3.1	3.2	3.2	11.8
No. 33	70.2	4.2	7.2	4.3	2.8	0.0	2.2	0.7	8.4
No. 34	70.1	3.8	4.2	7.4	0.7	0.0	3.2	1.9	8.7
TYPE IV									
No. 41	26.5	51.7	1.5	15.9	0.4	3.0	0.0	2.7	1.3
No. 42	30.2	54.2	0.9	2.5	0.0	3.1	0.4	0.9	7.8
No. 43	28.1	48.0	2.8	11.7	1.6	1.8	0.7	1.1	4.2
No. 43A	25.1	58.4	1.0	5.4	0.0	2.5	0.8	0.8	6.0
TYPE V									
No. 51	39.6	44.5	1.0	6.3	0.0	3.1	0.3	1.0	4.1

EXPERIMENTAL PROCEDURE

Heat of Hydration:

Portland cement when mixed with water hydrates rapidly for approximately 2 to 3 days during which interval a considerable quantity of heat is evolved. After about 3 days the rate of hydration subsides appreciably, and the rate of heat liberation is low during further periods of hydration.

The high rate of heat liberation during the early period of hydration permits the measurement of the heat of hydration of the cement by direct means, for which purpose a modification of the Carlson conduction calorimeter previously described by Lerch (**8**) is particularly applicable. With this apparatus a continuous record of the heat of hydration and also of the rate of heat liberation was obtained during the first 72 hr. of hydration. Studies of cement hydration at different temperature levels can also be performed conveniently with this apparatus. In the study reported by Lerch and Ford in Chapter 3, tests of the 27 cements were carried out at four different base temperatures, 40, 75, 90, and 105 F., in order to evaluate more fully the hydration characteristics of the cements.

All the cement pastes in the studies herein reported were prepared with a water-cement ratio of 0.40 by weight. Tests of pastes with higher water-cement ratios are in progress. The heats of hydration of the cements at later ages (up to 6½ yr.) were determined from the heats of solution of the various samples in an acid solvent. This method yields results that are independent of previous direct measurements of hydration and is based on Hess' law of constant heat summation. In accordance with this law the difference in the heats of solution of unhydrated and partially hydrated cement (the final states of the samples in the acid solvent are identical) represents the heat evolved during the hydration of the cement.

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[6] At the time these cements were manufactured there were no specifications for air-entraining cements. The cements in this investigation were interground with flake Vinsol resin and were designated as "treated" cements. In the present report they have been classified in accordance with their respective A.S.T.M. Designations (C 175), though they do not exactly meet the requirements of the current specification.

At the time these tests were begun there was no standard A.S.T.M. method of test for the heat of hydration at later ages. However, the procedure employed in these tests is essentially the same as the heat of solution method adopted in 1944 and described in A.S.T.M. Tentative Method of Test for Heat of Hydration of Portland Cement (C 186 – 44 T).[7] The only divergency of any consequence between the methods is that the final results of this study are expressed on the basis of the unignited cement weight rather than on the ignited cement weight basis.

In the present tests, the temperature rise within the calorimeter was calculated by linear extrapolation of the final rating period, a procedure which proved entirely satisfactory for the conditions under which the calorimeter was operated.

The apparatus employed in the tests consisted of a vinylite lined Dewar vacuum flask of 500-ml. capacity fitted with a carefully machined, hollow, lucite calorimeter heat, and containing a vinylite coated Beckmann thermometer and a lucite stirring assembly. The calorimeter, shown in detail in Fig. 6-1[5] was submerged in a water bath for temperature stability.

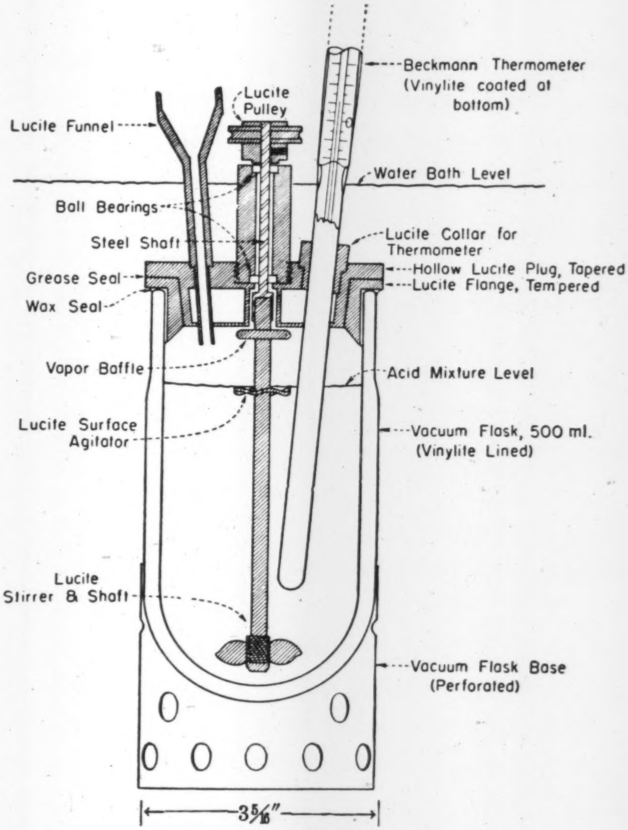
Lucite Pulley
Beckmann Thermometer (Vinylite coated at bottom)
Lucite Funnel
Water Bath Level
Ball Bearings
Steel Shaft
Lucite Collar for Thermometer
Hollow Lucite Plug, Tapered
Lucite Flange, Tempered
Grease Seal
Wax Seal
Vapor Baffle
Acid Mixture Level
Lucite Surface Agitator
Vacuum Flask, 500 ml. (Vinylite Lined)
Lucite Stirrer & Shaft
Vacuum Flask Base (Perforated)
3 5/16"

FIG. 6-1.—Heat of Solution Calorimeter.

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[7] 1944 Book of A.S.T.M. Standards, Part II, p. 1231. This method is now published as Standard Method C 186-49 and appears in 1949 Book of A.S.T.M. Standards, Part 3, p. 118.

Non-Evaporable Water and Vapor Sorption:

The non-evaporable water content and the water vapor sorption at 0.36 relative vapor pressure (r.v.p.) of a hydrated cement paste are measures of the extent of cement hydration in a manner analogous to heat of hydration. These corollary determinations were conducted at the different ages with the excess samples of hydrated pastes not required for the heat of solution tests. The non-evaporable water contents were determined in a manner similar to that described by Powers and Brownyard (9). The water vapor sorptions were determined in an unevacuated desiccator containing 50 per cent sulfuric acid.

TABLE 6-2.—AVERAGE HEATS OF HYDRATION OF THE DIFFERENT TYPES OF PORTLAND CEMENT AT EARLY AGES—CONDUCTION CALORIMETER.

Data in this table are from Table 3-15-A, Chapter 3, and appear also as part of Table 6-13 in the appendix to this report.

Type of Cement	Number of Cements Averaged	Average Heat of Hydration at Age and Temperature Indicated, cal. per g.			
		40 F.	75 F.	90 F.	105 F.
24 Hr.					
I and IA	12	18.5	43.7	52.3	60.9
II and IIA	6	14.9	34.7	39.9	47.8
III and IIIA	4	28.7	60.6	67.8	78.1
IV	4	15.3	31.9	36.3	40.5
V	1	15.9	37.6	37.7	43.0
48 Hr.					
I and IA	12	30.5	58.2	66.2	73.3
II and IIA	6	24.2	45.9	51.2	58.1
III and IIIA	4	44.1	75.1	80.4	87.3
IV	4	22.3	40.8	42.4	46.8
V	1	25.9	45.9	46.0	53.6
72 Hr.					
I and IA	12	36.9	68.0	73.9	80.0
II and IIA	6	28.6	53.6	57.1	64.3
III and IIIA	4	52.9	83.2	85.3	93.2
IV	4	25.7	46.6	45.8	51.2
V	1	31.5	50.8	51.4	61.5

The non-evaporable water content is defined as the water retained by the hydrated paste when dried over a desiccating mixture of magnesium perchlorate dihydrate and tetrahydrate and has been expressed as the percentage ratio of non-evaporable water to the unignited original cement content of the paste.

The water vapor sorption at 0.36 r.v.p. is that quantity of water adsorbed by the hydrated paste previously dried over the magnesium perchlorate desiccant. These results are also expressed on a percentage basis of the original unignited cement content of the paste.

The procedure employed in these determinations consisted of preparing a sample of the 30–150 mesh fraction of the ground hydrated paste in excess of that required for the heat of solution determination. A weighed sample of this material was dried to constant weight in a vacuum desiccator over the perchlorate mixture at 25 C., and the final weight obtained. The sample was then stored in a desiccator over 50 per cent sulfuric acid (0.36 r.v.p.) until a maximum weight was obtained. The sample was then ignited and its ignited weight determined. The non-evaporable water content and the vapor sorption were calculated from these data and the previously determined loss on ignition of the original cements.

EXPERIMENTAL RESULTS

Heat of Hydration at Early Ages (Conduction Calorimeter):

The heats of hydration of the cements at early ages are strongly influenced by the composition of the cement and temperature as shown in Table 6-2 and Fig. 6-2. In Table 6-2 only the average heat of hydration of the different types of cement, as affected by temperature and as a function of time, is shown. However, detailed data on the heat of hydration and rate of heat liberation of the individual cements at early ages and the averages of the different types of cement reproduced from Chapter 3 are included as an Appendix to this report.[8] The average results in Table 6-2 reveal the steady increase in the heat of hydration of the cement with age up to 3 days and the strong influence of temperature in accelerating the rate of hydration regardless of cement type. It may be noted that cement composition, as represented by the different types of cement, consistently manifests itself in the heat of hydration at the different ages and temperatures. This is strikingly revealed in Fig. 6-2. In this figure it may be noted that the higher temperatures appreciably accelerate the hydration of the different cements, and that at early ages the hydration of the type III cements at 40 F. is approximately equivalent to that of the type IV cements at 105 F.

The storage temperature of the cements in the conduction calorimeter by the very nature of the test departs from the selected base temperature in much the same manner as would the temperature of concrete under the usual conditions of concrete construction in which the heat is dissipated rapidly to the surroundings. This type of temperature history does not correspond to storage at a constant temperature, or to simulated "mass" storage as used in the heat of solution studies, and this fact should be kept in mind when comparisons are made between the results obtained by the different methods.

Heat of Hydration at Later Ages (Heat of Solution Method):

Neat cement pastes with a water-cement ratio of 0.40 by weight were prepared at 70 F., placed into small vials, and the vials sealed. One complete set of specimens was cured continuously at 70 F. until time of test, which is in accordance with the present A.S.T.M. Standard Method of Test for Heat of Hydration of Portland Cement (C 186 – 49).[9] A companion set of specimens was subjected to a simulated "mass" storage procedure in order more nearly to represent the temperature history of concrete in a large concrete structure. In this latter procedure the specimens were stored at 70 F. for one day, at 100 F. for the following 27 days, and at 70 F. thereafter until time of test. This procedure is similar to that described in the U. S. Bureau of Reclamation Specifications for Boulder Dam No. 591D, May 1933, and No. 566, March 1934.

The heats of hydration of the individual cements by the heat of solution

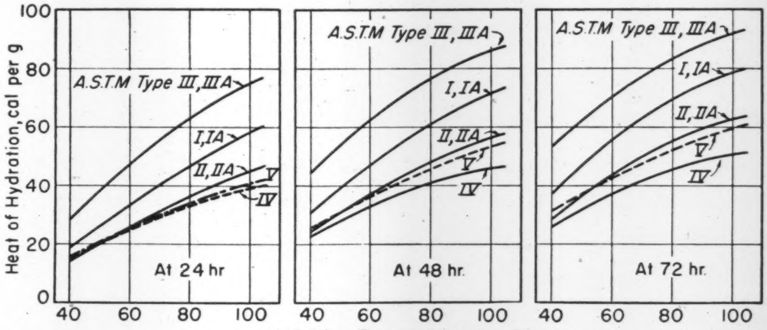

FIG. 6-2.—Effect of Temperature on the Heat of Hydration at Early Ages. Average Heat of Hydration of the Different Types of Portland Cement—Conduction Calorimeter.

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[8] See p. 1258.

[9] 1949 Book of A.S.T.M. Standards, Part 3, p. 118.

TABLE 6-3.—HEAT OF HYDRATION OF NEAT CEMENT PASTES BY THE HEAT OF SOLUTION METHOD.

w/c = 40 per cent by weight.

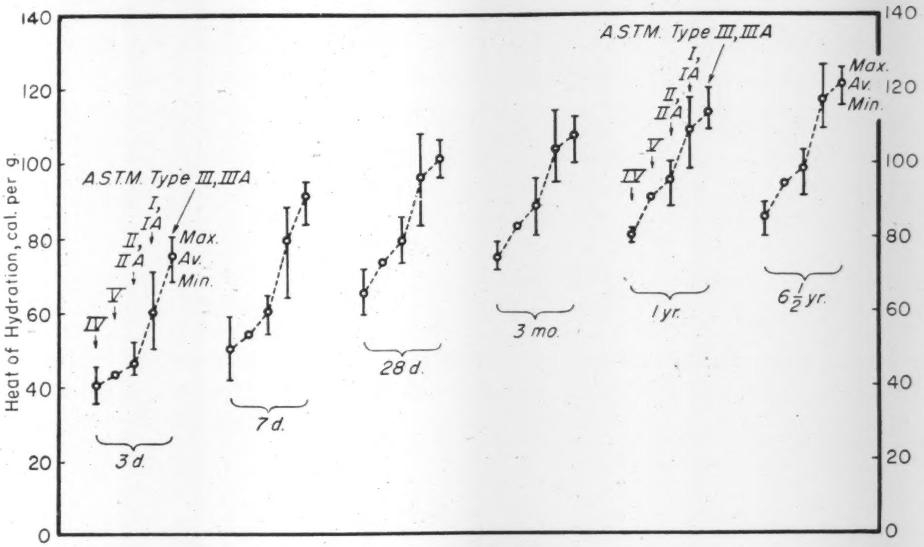
Cement	Heat of Hydration,[a] cal. per g. cement											
	Pastes Cured at 70 F.						Mass-Cured Pastes[b]					
	3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
TYPE I AND IA CEMENTS												
No. 11	62.5	81.0	94.2	104.4	110.1	114.0	81.3	91.6	98.3	102.1	104.2	112.7
No. 11T[c]	65.6	88.5	100.5	106.1	111.3	123.1	84.3	93.0	101.1	101.1	109.1	113.6
No. 12	57.7	83.2	99.7	107.2	114.6	123.3	79.2	93.9	100.1	101.3	107.2	117.4
No. 12T	55.2	78.4	98.4	109.4	111.6	120.9	78.5	90.6	97.6	98.9	107.8	117.7
No. 13	50.6	64.3	83.4	96.6	103.6	115.5	60.0	75.9	85.5	89.1	96.1	104.3
No. 14	63.8	78.0	86.2	95.4	98.4	110.1	73.3	79.6	85.3	86.4	94.1	102.7
No. 15	71.3	87.5	108.2	114.3	117.8	126.6	87.7	102.6	109.4	111.6	120.1	122.8
No. 16	60.2	79.3	91.3	98.0	104.2	112.1	72.6	80.4	87.5	90.6	104.8	105.0
No. 16T	58.7	73.8	88.9	95.1	102.7	109.5	73.2	79.6	88.8	92.8	100.0	106.9
No. 17	60.8	79.2	102.0	106.4	109.4	114.5	72.6	88.4	94.5	99.5	105.6	111.2
No. 18	60.6	81.2	99.9	107.7	110.7	118.3	75.9	89.4	99.5	102.1	108.8	117.7
No. 18T	61.1	82.2	102.3	106.9	109.8	117.6	81.9	90.6	101.9	104.3	111.0	119.9
TYPE II AND IIA CEMENTS												
No. 21	45.7	61.0	79.4	87.2	95.1	99.9	59.7	70.0	80.7	84.1	89.6	93.8
No. 21T	46.5	60.1	79.5	89.3	94.0	97.1	56.9	71.2	80.3	85.7	92.9	97.2
No. 22	43.6	62.7	85.8	96.0	100.6	103.2	57.9	72.8	83.8	88.4	96.9	99.4
No. 23	46.3	54.8	82.7	91.6	99.0	103.3	56.2	74.4	85.3	87.8	91.6	95.2
No. 24	52.1	64.9	76.8	85.1	93.7	94.4	61.1	68.6	76.6	82.0	86.3	91.7
No. 25	47.0	61.1	73.4	80.8	88.5	91.4	55.4	64.2	73.7	77.4	87.4	90.4
TYPE III AND IIIA CEMENTS												
No. 31	80.9	95.6	106.8	112.7	120.0	125.8	86.9	96.9	102.4	103.8	116.8	123.5
No. 33	78.0	92.2	101.5	107.8	113.5	120.5	89.2	95.8	101.0	102.2	109.3	116.1
No. 33T	78.0	94.5	100.7	108.6	112.8	122.9	90.3	96.3	100.8	101.7	107.6	117.1
No. 34	68.8	84.0	96.6	100.0	109.0	115.6	80.5	86.6	94.8	96.2	101.8	111.2
TYPE IV CEMENTS												
No. 41	44.9	52.9	65.8	73.6	78.6	84.2	49.1	56.0	65.9	67.8	74.4	78.8
No. 42	37.1	42.0	59.3	71.4	79.0	80.8	44.2	51.5	67.3	72.2	78.3	81.8
No. 43	45.8	59.1	71.9	78.8	82.4	89.5	52.2	61.0	71.9	73.5	83.2	88.7
No. 43A	35.9	46.2	65.3	74.0	82.5	86.7	44.3	53.5	70.6	73.7	81.5	88.9
TYPE V CEMENT												
No. 51	43.5	54.2	73.5	83.0	90.9	94.3	46.1	62.9	78.4	83.0	91.7	96.6

[a] Federal Specification SS-C-158a (1941) except results are expressed on dry unignited cement basis.

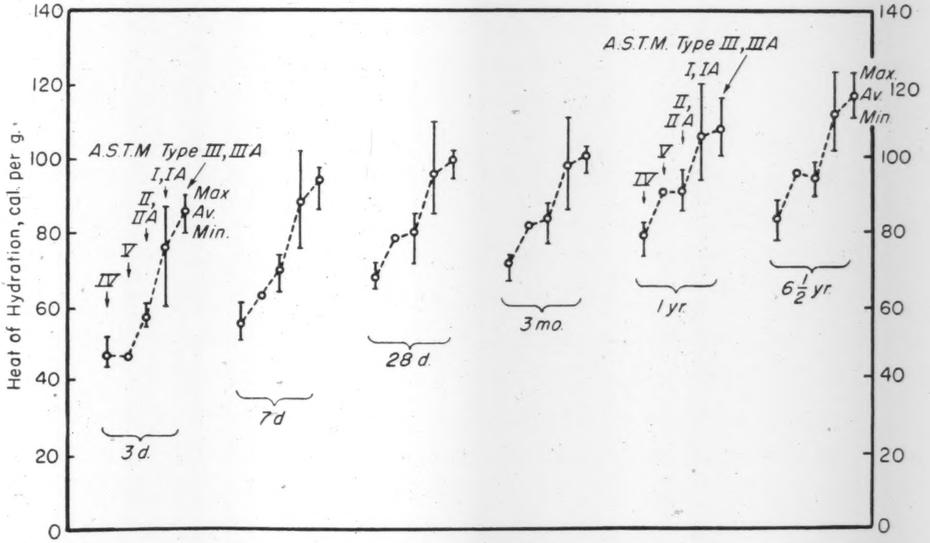
[b] Specimens stored at 70 F. for 1 day, at 100 F. for 27 days, and those used for tests at 3 months and 1 and 6½ yr. were stored at 70 F. from 28 days to time of test.

[c] Letter "T" after cement number indicates cements interground with flake Vinsol resin.

method for both curing procedures were determined at the ages of 3, 7, and 28 days, 3 months, 1 and 6½ yr. The essential data obtained in these tests are shown in Table 6-3 and Fig. 6-3. Here it may be noted that cement composition affects the heat of hydration at all ages up to 6½ yr. and that the higher storage temperature during the first 28 days in the mass-cured specimens increased the heat liberation at ages of 3 and 7 days. For 28 days and beyond, the mass-cured specimens show nearly the same or slightly lower values than those cured continuously at 70 F. Because of the large number of separate items of data for the different conditions, these trends are probably best revealed by comparisons of the average results for the various types of cement.



(*a*) Paste cured at 70 F.



(*b*) Paste mass cured.

FIG. 6-3.—Comparison of the Heats of Hydration of the Different Types of Cement.

Effect of Cement Composition and Fineness:

The effects of cement composition and fineness on the heat of hydration of the different cements is shown in Table 6-4. Included in this table are the average heats of hydration for the different cement types at the different ages and for both conditions of storage. It may be seen that under comparable conditions of test the average heats of hydration of the types I and III cements are considerably greater than those of the types II, IV, and V cements at all ages up to 6½ yr.

The type I cements on the average liberated about 25 to 30 cal. per g. more heat than did the type IV cements at 1 and 6½ yr., showing the strong and continued influence of cement composition on heat of hydration. The average type I cement in about 7 days had liberated as much heat as did the average type IV cement in 6½ yr.

TABLE 6-4.—AVERAGE HEATS OF HYDRATION OF DIFFERENT TYPES OF CEMENT—HEAT OF SOLUTION METHOD.

Cement Type	Number Averaged	Heat of Hydration at Age Indicated, cal. per g.											
		70 F. Storage						Mass Storage					
		3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
I and IA	12	60.7	79.7	96.3	103.9	108.7	117.1	76.7	87.9	95.8	98.3	105.7	112.7
II and IIA	6	46.8	60.8	79.6	88.3	95.2	98.2	57.9	70.2	80.1	84.2	90.8	94.6
III and IIIA	4	75.4	91.6	101.4	107.3	113.8	121.2	86.7	93.9	99.8	101.0	108.9	117.0
IV	4	40.9	50.1	65.6	74.5	80.6	85.3	47.5	55.5	68.9	71.8	79.4	84.5
V	1	43.5	54.7	73.5	83.0	90.9	94.2	46.1	62.9	78.4	83.0	91.7	96.6

Another type of comparison is made in Fig. 6-3. Figure 6-3(*a*) represents the data for the pastes cured continuously at 70 F. and includes the maximum, minimum, and average heat of hydration for each cement type at different ages. The results show that each cement type has a reasonably separate and distinct range in heat of hydration at the different ages and that, in general, only moderate overlapping occurs between the various types. The greatest range in the heats of hydration of cements of any particular type occurs among the type I cements, for which the specifications for chemical requirements are somewhat less restrictive than those of the other types of cement. The heats of hydration of types II, IV, and V cements are consistently below those of the types I and III cements at all ages.

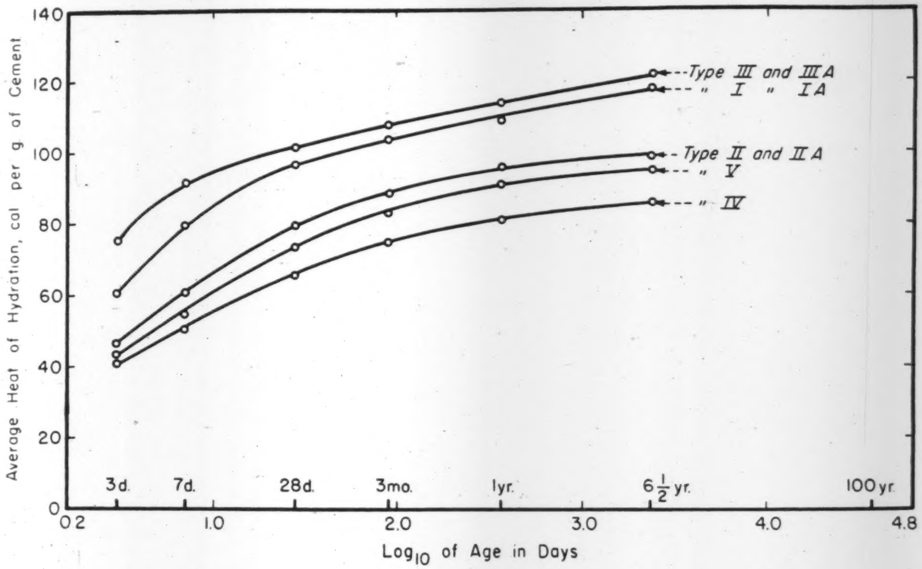
Figure 6-3(*b*) represents the tests of the cements subjected to "mass" curing procedure. It may be noted that the results shown in this figure are similar in character to those for the pastes cured at 70 F. in Fig. 6-3(*a*) although numerically different, particularly at the earlier ages.

The effects of cement composition are not only reflected in the total heat of hydration of the cement but also in the rate of heat liberation at the later ages. The increments of heat liberated by the different types of cement between the ages of 1 and 6½ yr. are shown in Table 6-5. The data are for pastes subjected to both continuous storage at 70 F., and simulated "mass" storage.

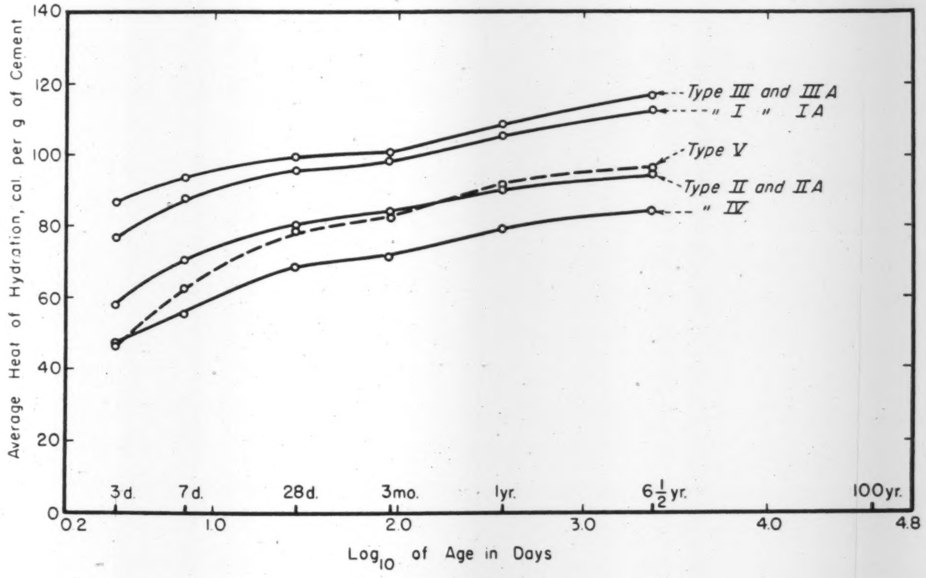
TABLE 6-5.—EFFECT OF CEMENT TYPE ON HEAT LIBERATION AT LATER AGES.

Cement Type	Number Averaged	Increase in Heat of Hydration from 1 yr. to 6½ yr., cal. per g.	
		70 F. Storage	Mass Storage
I and IA	12	8.4	7.0
III and IIIA	4	7.4	8.1
IV	4	4.7	5.2
V	1	3.3	4.9
II and IIA	6	3.1	3.8

Table 6-5 contains specific information on the question of the heat of hydration of the different types of cement at the later ages. It will be noted that types II, IV, and V cements liberate less heat than types I and III cements during the period from 1 to 6½ yr. From these data and



(*a*) Paste cured at 70 F.



(*b*) Paste mass cured.

Fig. 6-4.—Relationship Between Age and Average Heat of Hydration for Different Types of Cement—Heat of Solution Method. Water-cement ratio = 0.40 by weight.

from the curves in Fig. 6-4, it appears that the heat of hydration of the type IV and also of type II and type V cements will continue to be lower than that of the type I and type III cements even after much longer periods of curing. Additional hydrating paste samples are available for heat of hydration determinations at ages beyond 6½ yr.

Effect of Temperature:

The temperature at which the cement paste was cured affects the heat evolved by the cement. However, the cycle of varying temperatures used in the simulated mass storage of one set of cement pastes makes the direct isolation of the effect of temperature difficult. At 3 days the average heat of hydration of mass-cured pastes (stored for 1 day at 70 F. and 2 days at 100 F.) was approximately 20 per cent higher than the average for the pastes cured continuously at 70 F. At 7 days the heat of hydration of the mass-cured pastes was approximately 10 per cent higher than the average of the pastes cured at 70 F., whereas at 28 days the average heats of hydration of the pastes subjected to the two curing procedures were approximately the same. After 28 days the mass-cured specimens were returned to 70 F. storage and the average heats of hydration of these pastes was slightly below that of the pastes cured continuously at 70 F. when tested at 3 months and at 1 and 6½ yr. Under the conditions of these tests it appears that the initial impetus of increased temperature in accelerating the heat evolution of cement does not persist and has little effect on the heat of hydration at later ages.

Effect of Interground Vinsol Resin:

The heats of hydration observed for the non-air-entraining cements and the companion air-entraining cements containing interground Vinsol resin were about the same in most of the determinations. There was no consistent relationship among the small deviations that were observed, which indicates that the interground Vinsol resin had no significant effect on the heat of hydration of the cements.

Least Squares Analysis of Heat of Hydration Data:

The results of the heat of hydration tests were analyzed mathematically by the method of least squares to reveal the influence of cement composition on the heat of hydration of the different cements at the several ages. The least squares analyses were made on three different bases to determine which basis most satisfactorily fitted the experimental data. All three methods of calculation assume a linear and independent relationship between the composition of the cement and heat of hydration as follows:

Method 1–4 Variables:

$a(\%\ C_3S) + b(\%\ C_2S) + c(\%\ C_3A) + d(\%\ C_4AF) =$ heat of hydration, cal. per g. at a given age.

The compositions of the cements used in this method of analysis are calculated compound compositions as shown in Table 6-1-A.

Method 2–5 Variables:

$a(\%\ C_3S) + b(\%\ C_2S) + c(\%\ C_3A) + d(\%\ C_4AF) + e(\%\ SO_3) =$ heat of hydration, cal. per g. at a given age.

The calculated compound compositions were also used in this method of analysis. The SO_3 contents were determined by chemical analysis and are given in Table 6-1-A.

Method 3–6 Variables:

$a(\%\ C_3S) + b(\%\ C_2S) + c(\%\ C_3A +$ dark prismatic$) + d(\%\ C_4AF) + e(\%$ glass$) + f(\%$ undifferentiated$) =$ heat of hydration, cal. per g. at a given age

in which the compositions are the results of microscopic determinations on the various clinkers as reported in Table 6-1-B.

The six cements interground with Vinsol resin, and six additional cements (Nos. 13, 24, 31, 33, 34, and 41) having losses on ignition (Table 6-1-A) greater than an arbitrarily selected value of 1 per cent were not considered in any of the calculations. The interground Vinsol resin cements were omitted from the analyses on the basis that their consideration would automatically include repetition of certain compositions and would therefore tend to give undue weight to such compositions. It has been shown that cements having high ignition losses resulting from partial prehydration of the cement have significantly lower heats of hydration than corresponding cements of lower ignition loss (10). Accordingly, the cements with ignition losses greater than 1 per cent were omitted from the analyses in order to exclude any possible influence of preliminary hydration or carbonation. In addition, cement No. 21 was excluded from the analyses because of its low specific surface area which might affect its rate of hydration relative to the other cements included. The inclusion of this cement among those

TABLE 6-6.—RESULTS OF LEAST SQUARES ANALYSIS OF HEAT OF HYDRATION DATA.

Component	Coefficients of Linear Equations at Age Indicated, cal. per 0.01 g. of compound					
	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
PASTES CURED AT 70 F.—4 VARIABLES (METHOD 1)						
C_3S (Calculated)	0.58±0.08	0.53±0.11	0.90±0.07	1.04±0.05	1.17±0.07	1.17±0.07
C_2S (Calculated)	0.12±0.05	0.10±0.07	0.25±0.04	0.42±0.03	0.54±0.04	0.53±0.05
C_3A (Calculated)	2.12±0.28	3.72±0.39	3.29±0.23	3.11±0.17	2.79±0.23	3.28±0.25
C_4AF (Calculated)	0.69±0.27	1.18±0.37	1.18±0.22	0.98±0.16	0.90±0.22	1.11±0.24
PASTES CURED AT 70 F.—5 VARIABLES (METHOD 2)						
C_3S (Calculated)	0.54±0.08	0.51±0.11	0.91±0.07	1.07±0.04	1.22±0.04	1.20±0.07
C_2S (Calculated)	−0.01±0.09	0.04±0.13	0.29±0.07	0.53±0.04	0.74±0.04	0.65±0.08
C_3A (Calculated)	1.51±0.38	3.43±0.55	3.44±0.31	3.60±0.18	3.70±0.18	3.85±0.32
C_4AF (Calculated)	0.20±0.34	0.94±0.48	1.30±0.27	1.37±0.16	1.63±0.16	1.55±0.28
SO_3 (Chemical analysis)	8.92±4.12	4.34±5.94	−2.17±3.37	−7.16±1.91	−13.46±1.92	−8.28±3.44
PASTES CURED AT 70 F.—6 VARIABLES (METHOD 3)						
C_3S (Microscopic)	0.79±0.09	0.86±0.13	0.90±0.11	1.10±0.10	1.12±0.11	1.23±0.11
C_2S Microscopic)	0.28±0.06	0.30±0.09	0.39±0.07	0.52±0.07	0.59±0.08	0.67±0.07
C_3A + Dark prismatic (Microscopic)	2.98±0.38	4.47±0.49	3.07±0.42	2.67±0.40	2.43±0.43	3.38±0.41
C_4AF (Microscopic)	0.03±0.26	0.50±0.34	0.84±0.30	0.52±0.28	0.73±0.30	0.55±0.29
Glass (Microscopic)	0.78±0.18	1.42±0.24	0.58±0.20	0.75±0.19	1.28±0.21	0.79±0.20
Undifferentiated (Microscopic)	−0.93±0.46	−0.89±0.62	1.34±0.53	1.14±0.50	1.40±0.54	0.89±0.52
PASTES "MASS" CURED—4 VARIABLES (METHOD 1)						
C_3S (Calculated)	0.61±0.09	0.84±0.06	1.04±0.06	1.14±0.06	1.23±0.07	1.14±0.07
C_2S (Calculated)	0.10±0.06	0.14±0.04	0.45±0.04	0.52±0.04	0.60±0.04	0.68±0.04
C_3A (Calculated)	3.34±0.30	3.30±0.20	2.71±0.20	2.49±0.21	2.42±0.24	3.17±0.22
C_4AF (Calculated)	0.80±0.29	0.95±0.19	0.58±0.19	0.40±0.20	0.57±0.23	0.59±0.21
PASTES "MASS" CURED—5 VARIABLES (METHOD 2)						
C_3S (Calculated)	0.60±0.09	0.86±0.05	1.06±0.05	1.16±0.05	1.21±0.07	1.16±0.07
C_2S (Calculated)	0.07±0.11	0.24±0.06	0.54±0.06	0.62±0.06	0.54±0.08	0.74±0.08
C_3A (Calculated)	3.20±0.46	3.79±0.25	3.13±0.26	2.98±0.24	2.15±0.36	3.44±0.32
C_4AF (Calculated)	0.69±0.41	1.35±0.22	0.92±0.23	0.79±0.21	0.35±0.31	0.82±0.28
SO_3 (Chemical analysis)	2.02±4.98	−7.22±2.69	−6.29±2.84	−7.17±2.58	4.01±3.84	−4.08±3.49
PASTES "MASS" CURED—6 VARIABLES (METHOD 3)						
C_3S (Microscopic)	0.78±0.11	0.97±0.08	0.99±0.08	1.03±0.09	1.09±0.11	1.24±0.12
C_2S (Microscopic)	0.22±0.08	0.35±0.06	0.52±0.06	0.55±0.06	0.61±0.07	0.79±0.08
C_3A + Dark prismatic (Microscopic)	3.76±0.44	3.52±0.33	2.89±0.33	2.60±0.34	2.86±0.43	3.79±0.46
C_4AF (Microscopic)	0.37±0.30	−0.02±0.23	0.26±0.23	0.26±0.24	0.89±0.30	0.02±0.32
Glass (Microscopic)	1.59±0.21	0.85±0.16	1.28±0.16	1.43±0.16	1.03±0.20	1.26±0.22
Undifferentiated (Microscopic)	−0.11±0.54	0.41±0.41	0.91±0.42	1.05±0.43	0.87±0.54	0.15±0.57

mathematically studied would have broadened the range in the specific surface areas of the cements studied by approximately 45 per cent which might have more than offset the advantage of an additional item in the analysis. These omissions reduced the number of cements to 14. All 14 of these cements were subjected to the least squares analysis of method 3. However, two additional cements (Nos. 14 and 22) were excluded from the method 1 and method 2 analyses because the results of a preliminary partial method 1 analysis had indicated that these two cements alone accounted for more than half of the variance occurring in the results of the analysis.

Each of the three methods of least squares analysis was performed at each of the six different test ages for the pastes subjected to continuous curing at 70 F. and to the simulated mass curing procedure. The results of these analyses are shown in Table 6-6 which includes the numerical values for the heat coefficients of the individual cement components that best fit the experimental heat of hydration data. Also included are the probable errors of these values, these serving in each instance to establish the magnitude of error such that the probability of the error being greater is equal to the probability of the error being less than this amount. The results are also shown graphically in Fig. 6-5 in which the contributions of the individual components are expressed as calories per gram and in which the probable error is indicated.

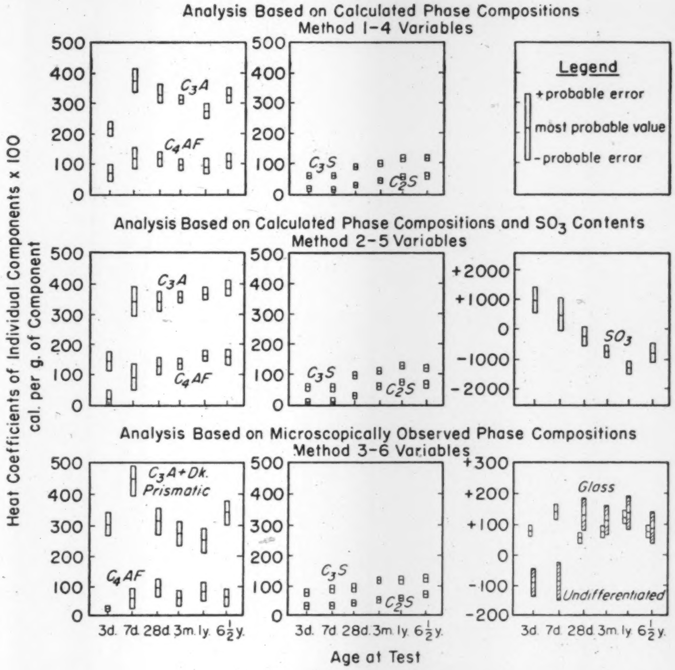
The results of the least squares analyses are only numerical expressions which most suitably reproduce the values for the heat of hydration of the individual cements included in the analyses, and may be influenced by the manner of preparation of the cements and by the presence of various minor constituents. The results may be directly applied to determine the presence of significant deviations among the particular cements studied and may be used in estimating the heats of hydration of cements other than those included in the analyses.

The numerical significance of the values resulting from a least squares analysis of this type is somewhat more difficult to interpret but may be indicative of various trends. The results of the present analysis indicate several significant trends that are of particular interest.

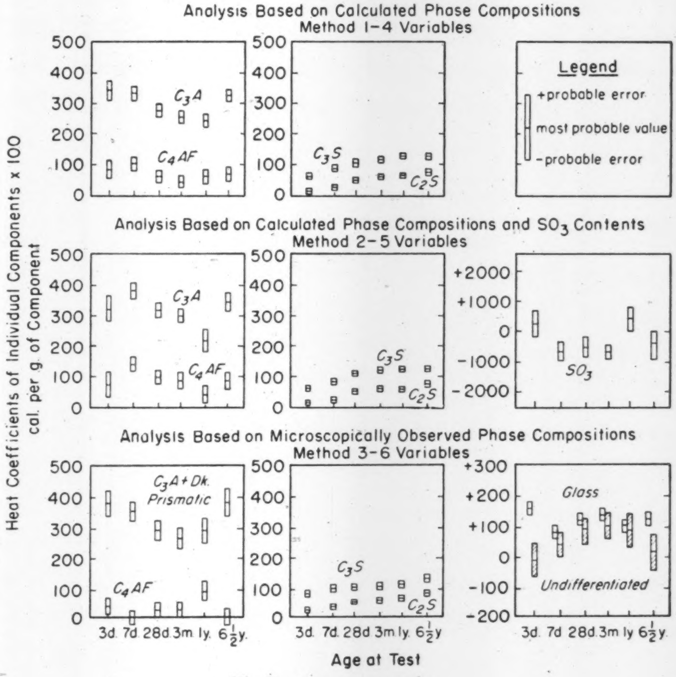
It may be observed in Fig. 6-5 that the derived heat coefficients of the calcium silicates steadily increase with increasing age. Regardless of the basis of analysis and the curing procedure, the 6½-yr. values obtained for either the C_3S or the C_2S agree satisfactorily among themselves and are also in good agreement with previously reported values (2, 3, 4, 11) for the ultimate heats of these compounds.

However, the coefficients derived for the aluminum compounds, C_3A and C_4AF, are decidedly different from previously reported values, this being particularly pronounced for the C_3A component for which the calculated heat coefficients are in general exceptionally high. The magnitude of the calculated heat coefficients of the aluminum compounds and the consistent increase in the coefficient for C_3A between 1 and 6½ yr. suggest that relatively slow secondary changes may have occurred during the hydration of these compounds.

The heat coefficients of the SO_3 content of the cements (method 2) are of particular interest in that they show a significant trend with age for the pastes cured continuously at 70 F. (Fig. 6-5(*a*)), whereas no significant trend is observed for the mass-cured pastes (Fig. 6-5(*b*)). Considering the probable error of the calculated results at the various ages, it appears that at 70 F., SO_3 performs a significant rôle in heat liberation. On the other hand, the data obtained for SO_3 in pastes sub-



(*a*) Paste cured at 70 F.



(*b*) Paste mass cured.

FIG. 6-5.—Mathematical Analysis of Heat of Hydration Data, Least Squares Method.

TABLE 6-7.—COMPARISON OF OBSERVED HEATS OF HYDRATION AND HEATS OF HYDRATION CALCULATED FROM THE RESULTS OF THE THREE METHODS OF LEAST SQUARES ANALYSIS.

Cement	Basis of Comparison	Heat of Hydration,[a] cal. per g. cement											
		Pastes Cured at 70 F.						Mass-Cured Pastes[b]					
		3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
TYPE I AND IA CEMENTS													
No. 11	Observed	62.5	81.0	94.2	104.4	110.1	114.0	81.3	91.6	98.3	102.1	104.2	112.7
	Calculated Method 1[c]	62.1	82.1	99.0	106.1	110.6	117.7	76.8	91.8	98.9	101.4	108.1	114.6
	Calculated Method 2[d]	60.4	81.6	99.6	107.2	112.4	117.9	77.7	92.5	99.3	101.2	107.4	115.2
	Calculated Method 3[e]	59.7	77.7	93.5	102.6	109.4	113.1	71.7	88.5	97.1	100.8	103.8	110.8
No. 11T[f]	Observed	65.6	88.5	100.5	106.1	111.3	123.1	84.3	93.0	101.1	101.1	109.1	113.6
	Calculated Method 1	62.6	80.9	99.9	107.0	111.4	118.6	79.5	92.5	99.4	102.1	108.6	115.5
	Calculated Method 2	61.0	82.0	100.3	108.2	113.8	120.1	79.1	93.8	100.5	103.4	107.9	116.2
	Calculated Method 3[g]	59.7	77.7	93.5	102.6	109.4	113.1	71.7	88.5	97.1	100.8	103.8	110.8
No. 12	Observed	57.7	83.2	99.7	107.2	114.6	123.3	79.2	93.9	100.1	101.3	107.2	117.4
	Calculated Method 1	60.0	82.0	96.3	105.1	109.5	116.7	78.2	90.2	97.7	100.2	106.9	114.3
	Calculated Method 2	58.3	80.9	98.5	107.3	113.3	119.5	77.6	91.8	99.2	101.9	105.3	115.8
	Calculated Method 3	60.9	81.2	99.1	105.1	109.8	117.8	78.1	91.5	98.6	100.7	106.8	114.9
No. 12T	Observed	55.2	78.4	98.4	109.4	111.6	120.9	78.5	90.6	97.6	98.9	107.8	117.7
	Calculated Method 1	61.0	79.5	97.9	105.2	109.6	116.9	78.1	90.1	97.7	100.3	106.8	114.8
	Calculated Method 2	58.7	80.8	98.5	107.0	113.1	119.0	77.6	91.9	99.3	102.1	105.7	115.8
	Calculated Method 3	60.9	81.2	99.1	105.1	109.8	117.8	78.1	91.5	98.6	100.7	106.8	114.9
No. 13	Observed	50.6	64.3	83.4	96.6	103.6	115.5	60.0	75.9	85.5	89.1	96.1	104.3
	Calculated Method 1	57.7	71.9	92.7	100.9	106.5	112.5	71.9	84.8	94.5	98.1	104.9	110.7
	Calculated Method 2	57.9	74.3	92.7	100.8	106.0	112.3	71.9	84.7	94.5	98.0	105.0	110.7
	Calculated Method 3	61.2	77.6	86.3	98.4	106.4	109.6	75.5	84.8	94.7	100.0	101.4	111.6
No. 14	Observed	63.8	78.0	86.2	95.4	98.4	110.1	73.3	79.6	85.3	86.4	94.1	102.7
	Calculated Method 1	52.0	64.2	84.4	92.6	98.2	103.6	63.8	75.7	85.8	89.1	96.2	101.8
	Calculated Method 2	52.4	67.2	84.3	92.0	97.5	103.2	63.9	75.3	85.6	88.7	96.5	101.6
	Calculated Method 3	55.3	70.4	86.4	94.3	99.5	106.2	65.5	80.7	86.0	88.8	98.1	102.6
No. 15	Observed	71.3	87.5	108.2	114.3	117.8	126.6	87.7	102.6	109.4	111.6	120.1	122.8
	Calculated Method 1	69.4	88.9	109.5	116.5	121.3	128.6	86.8	102.6	108.7	111.8	118.9	123.1
	Calculated Method 2	71.6	90.2	108.8	116.4	117.2	126.2	87.2	100.1	106.6	109.3	119.8	122.0
	Calculated Method 3	71.2	93.6	111.8	117.8	121.1	130.8	89.8	104.7	108.6	110.5	118.1	124.8
No. 16	Observed	60.2	79.3	91.3	98.0	104.2	112.1	72.6	80.4	87.5	90.6	104.8	105.0
	Calculated Method 1	56.7	70.8	91.0	98.5	104.4	110.0	68.3	82.8	91.6	94.9	102.7	105.1
	Calculated Method 2	57.2	71.2	90.8	97.8	103.0	109.2	68.4	81.5	90.7	93.8	102.9	104.9
	Calculated Method 3	63.0	80.5	91.7	100.3	105.2	112.5	74.3	84.7	91.5	89.5	104.2	108.8
No. 16T	Observed	58.7	73.8	88.9	95.1	102.7	109.5	73.2	79.6	88.8	92.8	100.0	106.9
	Calculated Method 1	56.7	69.6	91.3	98.8	104.6	110.4	68.8	82.9	91.6	95.0	102.5	106.2
	Calculated Method 2	58.7	72.6	90.8	97.2	101.7	108.5	69.3	81.2	90.3	93.4	103.4	105.3
	Calculated Method 3	63.0	80.5	91.7	100.3	105.2	112.5	74.3	84.7	91.5	89.5	104.2	108.8
No. 17	Observed	60.8	79.2	102.0	106.4	109.4	114.5	72.6	88.4	94.5	99.5	105.6	111.2
	Calculated Method 1	61.3	79.4	98.0	105.4	110.6	117.3	76.1	90.0	98.0	100.9	108.3	113.4
	Calculated Method 2	58.5	79.3	99.5	105.8	110.9	117.6	76.1	89.9	97.8	100.7	107.8	113.6
	Calculated Method 3	57.7	76.0	96.4	103.3	108.6	115.6	73.5	87.5	95.8	98.5	105.1	111.0
No. 18	Observed	60.6	81.2	99.9	107.7	110.7	118.3	75.9	89.4	99.5	102.1	108.8	117.7
	Calculated Method 1	61.0	83.4	98.8	106.0	110.1	117.6	79.5	91.4	98.5	100.9	107.3	115.5
	Calculated Method 2	60.5	81.4	98.9	106.2	110.3	117.7	79.4	91.1	98.8	100.7	107.2	115.8
	Calculated Method 3	63.6	81.0	96.8	105.8	112.5	118.2	77.2	91.4	97.9	100.8	107.2	115.3
No. 18T	Observed	61.1	82.2	102.3	106.9	109.8	117.6	81.9	90.6	101.9	104.3	111.0	119.9
	Calculated Method 1	61.3	80.6	98.3	105.6	109.7	117.2	79.0	90.6	97.9	100.4	106.7	115.5
	Calculated Method 2	59.9	82.4	98.7	106.7	111.8	118.5	78.7	91.7	99.0	101.5	106.0	116.1
	Calculated Method 3	63.6	81.0	96.8	105.8	112.5	118.2	77.2	91.4	97.9	100.8	107.2	115.3
TYPE II AND IIA CEMENTS													
No. 21	Observed	45.7	61.0	79.4	87.2	95.1	99.9	59.7	70.0	80.7	84.1	89.6	93.8
	Calculated Method 1	48.1	56.8	79.0	88.5	95.6	100.1	57.5	69.4	82.7	86.6	94.5	99.7
	Calculated Method 2	44.0	58.5	80.1	91.7	101.8	103.9	56.6	72.7	85.6	89.9	92.6	101.6
	Calculated Method 3	45.9	57.9	75.9	85.0	91.6	96.0	54.7	66.4	78.2	81.7	89.5	94.2
No. 21T	Observed	46.5	60.1	79.5	89.3	94.0	97.1	56.9	71.2	80.3	85.7	92.9	97.2
	Calculated Method 1	47.3	56.5	77.6	86.6	93.3	97.9	56.9	68.2	80.7	84.3	91.9	97.3
	Calculated Method 2	43.5	58.2	78.6	89.6	99.0	101.4	56.0	71.2	83.4	87.3	90.2	99.1
	Calculated Method 3	45.9	57.9	75.9	85.0	91.6	96.0	54.7	66.4	78.2	81.7	89.5	94.2

[a] to [g] See p. 1252.

TABLE 6-7.—*Continued.*

Cement	Basis of Comparison	Heat of Hydration,[b] cal. per g. cement											
		Pastes Cured at 70 F.						Mass-Cured Pastes[b]					
		3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
Type II and IIA Cements—*Continued*													
No. 22	Observed	43.6	62.7	85.8	96.0	100.6	103.2	57.9	72.8	83.8	88.4	96.9	99.4
	Calculated Method 1	49.9	60.6	81.5	89.4	95.6	100.8	60.0	72.2	82.6	85.7	93.5	98.1
	Calculated Method 2	47.1	62.3	82.2	91.6	99.8	103.3	59.4	74.4	84.6	88.0	92.2	99.4
	Calculated Method 3	44.6	57.5	81.3	88.8	95.4	98.8	55.9	74.8	80.4	83.9	92.2	93.3
No. 23	Observed	46.3	54.8	82.7	91.6	99.0	103.3	56.2	74.4	85.3	87.8	91.6	95.2
	Calculated Method 1	51.5	62.6	83.8	91.0	101.6	102.8	59.2	74.2	83.4	86.4	95.6	95.9
	Calculated Method 2	49.0	61.8	84.4	92.6	100.5	104.3	58.7	75.3	84.5	87.6	94.4	96.9
	Calculated Method 3	49.2	62.4	87.5	96.5	103.3	106.2	62.2	76.5	87.7	91.9	98.3	100.3
No. 24	Observed	52.1	64.9	76.8	85.1	93.7	94.4	61.1	68.6	76.6	82.0	86.3	91.7
	Calculated Method 1	48.7	59.5	79.6	86.3	92.0	97.3	57.7	70.1	78.6	81.1	89.0	92.5
	Calculated Method 2	48.7	62.1	79.6	86.2	92.0	97.2	57.7	70.1	78.6	81.0	89.1	92.5
	Calculated Method 3	57.3	75.6	70.1	89.3	94.5	101.7	65.8	67.3	79.2	81.2	97.5	95.6
No. 25	Observed	47.0	61.1	73.4	80.8	88.5	91.4	55.4	64.2	73.7	77.4	87.4	90.4
	Calculated Method 1	44.4	56.9	73.6	81.2	87.5	92.2	52.2	63.8	74.2	76.8	85.1	89.0
	Calculated Method 2	44.7	57.0	73.8	81.1	87.0	91.9	52.4	63.1	73.7	76.4	85.0	88.9
	Calculated Method 3	47.2	60.8	73.3	80.7	87.0	92.6	52.9	60.6	72.1	74.9	88.7	89.1
Type III and IIIA Cements													
No. 31	Observed	80.9	95.6	106.8	112.7	120.0	125.8	86.9	96.9	102.4	103.8	116.8	123.5
	Calculated Method 1	61.5	77.5	98.0	105.5	110.5	116.9	76.9	90.9	98.6	102.0	108.5	113.6
	Calculated Method 2	67.7	82.0	96.5	100.5	101.2	111.2	78.3	85.8	94.3	97.0	111.3	110.8
	Calculated Method 3	65.8	96.2	102.1	109.9	113.8	112.0	83.9	97.7	102.9	105.7	109.7	118.0
No. 33	Observed	78.0	92.2	101.5	107.8	113.5	120.5	89.2	95.8	101.0	102.2	109.3	116.1
	Calculated Method 1	63.4	79.6	100.8	108.0	113.0	119.6	78.6	93.6	100.6	104.0	110.8	115.0
	Calculated Method 2	70.3	84.1	99.1	102.4	102.7	113.2	80.2	88.0	95.8	98.5	113.9	111.8
	Calculated Method 3	78.5	101.0	110.4	117.9	119.9	132.0	92.3	107.8	109.1	110.5	118.7	129.3
No. 33T	Observed	78.0	94.5	100.7	108.6	112.8	122.9	90.3	96.3	100.8	101.7	107.6	117.1
	Calculated Method 1	61.7	77.6	98.4	105.7	110.8	117.2	76.8	91.1	98.6	102.0	108.7	113.4
	Calculated Method 2	67.5	81.8	97.0	101.1	102.2	111.9	78.1	86.4	94.6	97.4	111.3	110.7
	Calculated Method 3	78.5	101.0	110.4	117.9	119.9	132.0	92.3	107.8	109.1	110.5	118.7	129.3
No. 34	Observed	68.8	84.0	96.6	100.0	109.0	115.6	80.5	86.6	94.8	96.2	101.8	111.2
	Calculated Method 1	57.1	67.0	91.1	98.8	105.4	110.2	67.0	83.4	92.3	96.5	104.3	104.4
	Calculated Method 2	60.6	69.7	90.3	95.9	100.2	106.9	67.8	80.5	89.9	93.7	105.8	102.8
	Calculated Method 3	62.9	79.3	97.4	105.9	109.8	116.8	75.5	89.7	95.1	98.1	106.8	109.7
Type IV Cements													
No. 41	Observed	44.9	52.9	65.8	73.6	78.6	84.2	49.1	56.0	65.9	67.8	74.4	78.8
	Calculated Method 1	37.5	45.8	63.7	71.3	77.3	81.9	44.5	53.0	64.4	66.5	74.4	80.9
	Calculated Method 2	37.9	50.5	63.7	71.0	76.8	81.5	44.6	52.7	64.2	66.2	74.6	80.7
	Calculated Method 3	42.3	57.6	66.7	73.1	81.7	85.6	49.8	50.0	67.6	70.4	84.1	85.2
No. 42	Observed	37.1	42.0	59.3	71.4	79.0	80.8	44.2	51.5	67.3	72.2	78.3	81.8
	Calculated Method 1	35.2	42.4	59.5	70.4	78.6	81.2	40.3	49.8	67.2	71.5	79.4	84.1
	Calculated Method 2	34.6	42.2	59.9	71.1	79.5	81.8	40.1	49.8	67.1	71.6	78.8	84.4
	Calculated Method 3	36.6	44.8	65.4	76.4	85.4	86.9	43.6	57.0	72.2	76.6	80.6	88.7
No. 43	Observed	45.8	59.1	71.9	78.8	82.4	89.5	52.2	61.0	71.9	73.5	83.2	88.7
	Calculated Method 1	42.8	57.3	71.4	79.2	85.1	90.2	51.8	61.3	72.4	74.4	82.5	88.9
	Calculated Method 2	44.1	58.2	71.3	78.2	82.8	88.8	52.2	59.8	71.1	73.0	82.8	88.3
	Calculated Method 3	46.3	62.8	74.0	79.8	87.8	92.8	55.7	62.3	74.5	76.8	88.3	92.6
No. 43A	Observed	35.9	46.2	65.3	74.0	82.5	86.7	44.3	53.5	70.6	73.7	81.5	88.9
	Calculated Method 1	40.6	51.1	67.8	77.9	85.3	87.0	48.1	58.0	73.4	76.9	85.0	90.8
	Calculated Method 2	41.7	51.9	67.7	77.0	83.5	88.0	48.4	56.5	72.0	75.9	85.2	90.3
	Calculated Method 3	37.6	44.3	62.3	72.2	80.2	83.3	41.4	52.3	68.0	71.8	77.9	85.2
Type V Cements													
No. 51	Observed	43.5	54.2	73.5	83.0	90.9	94.3	46.1	62.9	78.4	83.0	91.7	96.6
	Calculated Method 1	43.1	51.1	70.9	80.6	88.4	91.7	49.3	61.6	76.1	80.2	88.5	90.8
	Calculated Method 2	41.9	50.6	71.3	81.5	90.0	92.7	49.0	62.1	76.4	80.9	87.7	91.5
	Calculated Method 3	45.1	55.4	68.6	79.7	87.0	91.1	51.1	61.4	73.6	78.3	85.3	92.6

(Continued on page 1252.)

TABLE 6-7.—*Concluded*

Number of Cements Analyzed	Calculated Method	Probable Error (Plus or Minus), cal. per g. cement											
		Pastes Cured at 70 F.						Mass-Cured Pastes					
		3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
12	1	2.44	3.38	1.98	1.49	1.99	2.16	2.66	1.70	1.75	1.85	2.07	1.97
12	2	2.27	3.25	1.85	1.05	1.05	1.89	2.73	1.48	1.54	1.41	2.12	1.93
14	3	2.74	3.66	3.13	2.97	3.18	3.05	3.19	2.39	2.45	2.52	3.16	3.36

[a] Federal Specification SS-C-158a (1941) except results are expressed on dry unignited cement basis.

[b] Specimens stored at 70 F. for 1 day, at 100 F. for 27 days, and those used for tests at 3 months and 1 and 6½ yr. were stored at 70 F. from 28 days to time of test.

[c] *Method 1*, four variables, analysis based on calculated phase compositions.

[d] *Method 2*, five variables, analysis based on calculated phase compositions and SO_3 contents.

[e] *Method 3*, six variables, analysis based on microscopically observed phase compositions.

[f] Letter "T" after cement number indicates cements interground with flake Vinsol resin.

[g] The compositions of the clinkers interground with flake Vinsol resin used in applying Method 3 are those of the corresponding clinkers not interground with flake Vinsol resin.

jected to a temperature of 100 F. during the early period of the "mass" curing procedure show no consistent trend at the various ages of test.

The most probable values obtained for the heat coefficients of the various cement components (Table 6-6) resulting from the least squares analyses were used to calculate the heats of hydration of all the cements in this study. A comparison of the observed heats of hydration and the heats of hydration calculated from the results of the three different methods of least squares analysis is shown in Table 6-7. This table includes the results for all 27 cements for both methods of curing and at all the ages and in addition includes the probable error (12) indicated by the degree of fit between the observed and calculated heats of hydration of the cements actually subjected to the least squares analyses. As shown in Table 6-7 there is, in general, reasonably satisfactory agreement between the observed and calculated heats of hydration. However there is no consistent trend between the degree of agreement obtained between the observed heat of hydration and that calculated on the basis of either the calculated or microscopically observed phase compositions of the cements. In certain instances the heats of hydration calculated from the least squares analyses on the basis of the calculated compositions (methods 1 and 2) most nearly reproduce the observed heats of hydration; in other instances, more satisfactory agreement is obtained on the basis of the microscopically observed phase compositions (method 3). The relative merits of the three different bases of expression for the heat of hydration are probably best indicated by the calculated probable errors shown in Table 6-7. Although there is considerable variation in the probable errors calculated for the different ages and the two curing procedures for any particular method of least squares analysis, the average probable errors for all the test conditions for the individual methods were as follows: method 1, 2.12 cal. per g.; method 2, 1.88 cal. per g.; and method 3, 2.98 cal. per g. It would therefore appear that method 2, based on the calculated phase compositions and the SO_3 contents better fits the data for the cements from which the coefficients were calculated than do either method 1, based on the calculated phase compositions without the SO_3 contents, or method 3, which was based on the microscopically observed phase compositions of the clinkers.

The percentage of each of the calculated phase compounds in cement is a linear function of the percentages of the various oxides (SiO_2, Al_2O_3, Fe_2O_3, and CaO), after correction of the calcium oxide for free lime and gypsum. Therefore, the coefficients obtained by the

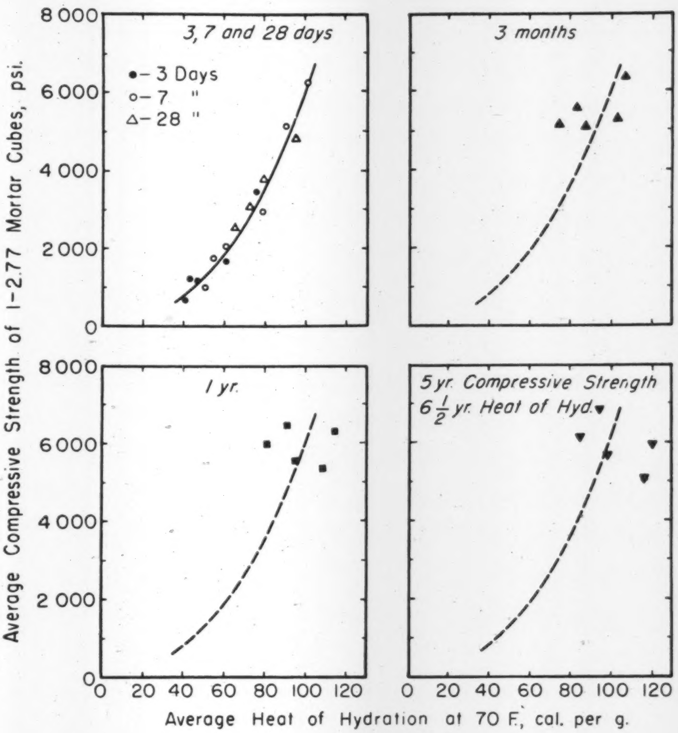


FIG. 6-6.—Relationship Between the Average Compressive Strength and the Average Heat of Hydration of the Five Types of Non-Air-Entraining Cement at Various Ages.

TABLE 6-8.—AVERAGE COMPRESSIVE STRENGTHS OF MORTAR CUBES AND HEATS OF HYDRATION OF THE DIFFERENT TYPES OF NON-AIR-ENTRAINING PORTLAND CEMENTS AT VARIOUS AGES.

Cement Type	Number Averaged	Compressive Strength[a] at Age Indicated, psi.						Heat of Hydration (at 70 F.) at Age Indicated, cal. per g.					
		3 day	7 day	28 day	3 month	1 yr.	5 yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
I	8	1689	2944	4789	5279	5325	5076	60.9	79.2	95.6	103.7	108.6	116.8
II	5	1180	2034	3728	5094	5520	5692	46.9	60.9	79.6	88.1	95.4	98.4
III	3	3457	5103	6250	6367	6300	5923	75.9	90.6	101.6	106.8	114.2	120.6
IV	4	688	1000	2510	5110	5980	6100	40.9	50.1	65.6	74.5	80.6	85.3
V	1	1240	1780	3010	5550	6460	6880	43.5	54.7	73.5	83.0	90.9	94.2

[a] 1–2.77 graded Ottawa sand mortar 2-in. cubes continuously cured under water at 70 F.—strength data from Table 3-11-C Chapter 3.

method of least squares based on the calculated phase compositions could also have been derived with equal precision had the analysis been based on the actual chemical oxide composition of the cements after correction of the calcium oxide (2).

Compressive Strength and Heat of Hydration:

Average compressive strengths and heats of hydration for the different types of non-air-entraining cements are given in Table 6-8. Figure 6-6 shows relationships at the different ages between the average compressive strengths of 2-in. mortar cubes prepared from the different types of non-air-entraining cements and the average heats of hydration of the same cements cured at the same temperature (70 F.). It may be noted that a definite relationship exists between strength and heat of hydration at the ages of 3, 7, and 28 days, but also that there is no such relationship at the ages of 3 months, 1 and 5 yr. For comparison, the curve for the early ages is repeated on the other three diagrams of Fig. 6-6.

As previously mentioned, it might be anticipated from the results of strength tests that the type IV cements, for example, would liberate more heat at later ages than type I cements. However, the data in Table 6-8 and Fig. 6-6 indicate that at the later ages the compressive strengths bear little relationship to heat of hydration. All of the different types of cement continued to liberate additional heat during the interval from 1 to 5 yr., but, as may be seen in Table 6-8,

TABLE 6-9.—NON-EVAPORABLE WATER CONTENTS AND VAPOR SORPTIONS.
Neat cement pastes, w/c = 40 per cent by weight.

Cement	Pastes Cured at 70 F.										Mass-Cured Pastes											
	Non-Evaporable Water, per cent by weight of Cement					Sorption at 0.36 r.v.p., per cent by weight of Cement					Non-Evaporable Water, per cent by weight of Cement						Sorption at 0.36 r.v.p., per cent by weight of Cement					
	7 day	28 day	3 month	1 yr.	6½ yr.	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
TYPE I AND IA CEMENTS																						
No. 11	15.7	18.0	19.7	21.0	22.7	5.0	6.0	6.9	7.1	7.8	15.3	18.1	18.9	19.6	20.3	20.7	5.3	5.7	5.9	6.6	6.8	6.8
No. 11T[a]	16.5	19.4	20.2	21.0	23.2	5.9	6.4	7.0	7.2	7.7	15.5	17.8	19.6	20.0	21.1	23.1	5.7	6.1	6.2	7.1	7.3	7.8
No. 12	16.2	19.2	20.8	21.8	24.0	4.9	6.7	7.4	7.5	8.1	15.0	17.7	19.6	20.0	21.1	23.3	5.2	6.0	6.3	7.2	7.4	7.7
No. 12T	14.8	19.4	20.9	21.9	23.7	6.1	6.5	6.8	7.5	7.9	15.6	17.7	19.7	20.2	21.8	23.6	5.3	6.1	6.3	7.2	7.5	7.9
No. 13	11.9	15.4	18.0	19.3	21.0	4.0	5.1	5.8	6.8	7.7	11.3	13.8	17.1	17.7	19.1	21.1	3.9	4.7	5.5	6.3	6.6	7.2
No. 14	14.8	17.7	18.3	20.0	22.2	5.1	5.4	6.4	7.0	7.2	14.0	15.1	17.2	17.6	18.9	21.9	5.0	5.4	5.8	6.5	6.7	7.3
No. 15	17.3	20.1	21.6	21.8	24.1	4.4	6.6	7.0	7.5	7.9	16.1	20.2	21.1	21.4	22.2	23.8	5.0	5.7	6.8	7.5	7.5	7.2
No. 16	14.9	17.3	18.9	19.7	21.4	3.9	5.7	6.3	6.6	7.2	13.8	16.2	17.5	18.0	20.0	21.8	4.5	4.7	5.6	6.2	6.6	6.6
No. 16T	14.9	16.6	18.2	19.6	21.0	3.5	5.5	6.0	7.6	7.2	13.6	15.9	17.4	17.9	19.5	21.8	4.4	4.6	5.6	6.2	6.5	6.7
No. 17	14.7	18.6	20.0	20.4	22.0	4.3	6.3	7.1	7.0	7.7	14.9	17.3	18.9	19.4	20.7	22.5	4.3	5.5	6.1	7.2	7.0	7.0
No. 18	16.2	19.2	20.9	21.7	22.6	4.8	6.5	7.6	7.4	7.6	16.0	17.9	19.8	20.5	21.5	22.6	4.8	5.9	6.4	7.6	7.4	8.0
No. 18T	17.0	20.2	21.0	21.7	22.4	4.7	6.8	7.7	7.7	7.9	16.5	18.2	20.2	21.0	21.5	22.1	5.0	6.1	6.7	7.7	7.6	7.9
TYPE II AND IIA CEMENTS																						
No. 21	11.9	15.0	17.5	18.5	20.0	3.6	5.5	6.3	6.9	6.3	11.8	14.2	16.4	17.1	18.5	19 8	3.5	4.7	5.6	6.8	6.9	7.3
No. 21T	12.0	15.1	17.5	18.7	20.4	3.7	5.6	6.5	7.2	7.6	12.1	14.4	16.8	17.6	18.9	20.5	3.6	4.9	5.9	7.1	7.3	7.7
No. 22	12.9	16.4	18.6	20.2	21.4	3.4	5.9	6.6	6.3	7.4	13.1	15.2	17.5	18.2	19.1	20.9	3.4	5.1	5.9	7.0	6.9	7.2
No. 23	11.3	15.8	...	19.5	20.0	3.6	5.5	...	6.0	6.8	11.5	14.9	16.6	17.1	18.3	19.1	3.3	4.7	5.5	6.5	6.5	7.0
No. 24	12.4	15.0	...	18.6	20.0	4.2	5.3	...	5.7	6.6	12.0	14.0	15.0	15.6	17.6	18.0	3.6	4.2	5.0	5.8	6.4	6.6
No. 25	12.1	15.0	...	17.5	19.7	3.9	5.2	...	5.7	5.8	11.6	13.6	15.5	16.0	17.7	18.2	3.2	4.3	5.1	6.0	6.1	6.7
TYPE III AND IIIA CEMENTS																						
No. 31	17.9	20.2	...	23.1	23.9	5.7	6.5	...	6.1	7.0	17.4	19.1	20.4	21.0	21.3	23.0	5.3	6.2	6.3	6.6	6.6	7.1
No. 33	17.7	19.5	...	21.6	22.6	5.6	6.4	...	6.1	6.8	17.3	18.4	19.6	20.1	21.1	21.8	5.2	6.0	6.1	6.4	6.6	7.2
No. 33T	17.8	19.3	...	22.1	23.4	5.7	6.3	...	6.3	6.9	17.7	18.7	19.9	20.6	21.2	22.5	5.4	6.2	6.5	6.7	6.8	7.2
No. 34	16.2	17.9	...	21.1	21.9	5.3	6.4	...	5.9	6.6	15.4	16.7	18.3	18.9	19.8	22.2	4.9	5.6	5.8	6.2	6.2	6.5
TYPE IV CEMENTS																						
No. 41	11.1	13.2	...	18.0	19.2	3.7	5.1	...	5.7	6.5	10.0	11.5	14.3	15.5	17.1	18.5	2.9	3.9	5.1	5.8	6.1	6.5
No. 42	8.5	11.9	16.5	18.8	19.8	3.3	4.8	...	6.2	7.0	8.7	10.5	14.8	16.6	17.6	18.9	2.7	3.6	5.6	6.4	6.6	6.7
No. 43	12.1	13.9	16.2	18.5	19.6	3.7	4.6	6.1	5.9	6.9	11.4	12.6	15.1	16.3	17.7	19.4	3.6	4.2	5.2	5.9	6.3	6.6
No. 43A	9.7	12.5	15.9	18.4	19.6	3.2	4.6	6.4	6.2	6.8	9.2	11.4	15.3	16.4	17.6	19.6	3.1	3.9	5.6	6.2	6.6	6.9
TYPE V CEMENT																						
No. 51	11.0	13.6	16.7	19.6	20.7	3.8	5.0	6.4	6.3	7.1	10.6	13.0	16.2	17.3	18.3	20.0	3.5	4.3	5.7	6.4	6.6	6.8

[a] Letter "T" after cement number indicates cements interground with flake Vinsol resin.

the average strengths of mortar cubes for the types I and III cements actually showed a slight decrease during this period. This is a further indication of the impracticability of attempting to estimate the heat of hydration of cements from strength data at ages beyond 28 days.

TABLE 6-10.—AVERAGE NON-EVAPORABLE WATER CONTENTS AND VAPOR SORPTIONS OF THE DIFFERENT TYPES OF CEMENT.

Cement Type	Number Averaged	Non-Evaporable Water, per cent by weight of cement						Sorption at 0.36 r.v.p., per cent by weight of cement					
		3 day	7 day	28 day	3 month	1 yr.	6½ yr.	3 day	7 day	28 day	3 month	1 yr.	6½ yr.
PASTES CURED AT 70 F.													
I and IA	12	...	15.4	18.4	19.9	20.8	22.5	...	4.8	6.1	6.8	7.2	7.7
II and IIA	6	...	12.1	15.4	...	18.8	20.2	...	3.7	5.5	...	6.3	6.7
III and IIIA	4	...	17.4	19.2	...	22.0	22.9	...	5.6	6.4	...	6.1	6.8
IV	4	...	10.3	12.9	...	18.4	19.5	...	3.5	4.8	...	6.0	6.8
V	1	...	11.0	13.6	16.7	19.6	20.7	...	3.8	5.0	6.4	6.3	7.1
MASS-CURED PASTES													
I and IA	12	14.8	17.2	18.9	19.4	20.6	22.4	4.9	5.5	6.1	6.9	7.1	7.3
II and IIA	6	12.0	14.4	16.3	16.9	18.3	19.4	3.4	4.7	5.5	6.5	6.7	7.1
III and IIIA	4	16.9	18.2	19.5	20.1	20.8	22.4	5.2	6.0	6.2	6.5	6.6	7.0
IV	4	9.8	11.5	14.9	16.2	17.5	19.1	3.1	3.9	5.4	6.1	6.4	6.7
V	1	10.6	13.0	16.2	17.3	18.3	20.0	3.5	4.3	5.7	6.4	6.6	6.8

TABLE 6-11.—COMPARISONS OF HEAT OF HYDRATION AND NON-EVAPORABLE WATER, AND SORPTION AND NON-EVAPORABLE WATER.

Cement Type	Average Heat of Hydration, cal. per g. for each 1 per cent of non-evaporable water		Average per cent Adsorption (0.36 r.v.p.) for each 1 per cent of non-evaporable water	
	70 F. Cured	Mass-Cured	70 F. Cured	Mass-Cured
III and IIIA	5.25	5.13	0.307	0.318
I and IA	5.20	5.10	0.331	0.333
II and IIA	5.03	4.90	0.333	0.343
V	4.89	4.78	0.344	0.347
IV	4.67	4.61	0.345	0.350

Non-Evaporable Water Contents and Vapor Sorptions:

The non-evaporable water contents and the water vapor sorptions at 0.36 relative vapor pressure of the individual cements are shown in Table 6-9.

The non-evaporable water content and the water sorption of the cement pastes, like the heat of hydration, are measures of the extent of cement hydration. It may be noted that there are some variations in these factors among the different cement types, as was also the case in the heats of hydration. Both the non-evaporable water and water sorption values, like those for heats of hydration, continued to increase with duration of paste curing.

The average results at the different ages for the individual types of cement subjected to the two storage conditions are shown in Table 6-10. Here it can be observed that significant differences occur among the several cement types, particularly at the earlier ages. The average non-evaporable water contents for types II, IV, and V cements are significantly lower than the average for types I and III cements at all ages studied.

General relationships exist between heat of hydration and either the non-evaporable water content or water sorption as is shown in Fig. 6-7. It may be noted that the non-evaporable water content, the vapor sorption, and the heat of hydration of the paste are, to a degree, all measures of the extent of hydration of the cement.

These general relationships appear to be influenced somewhat by cement composition but such variations as occur are not excessive. However, these variations may be of importance in detailed studies involving these factors and cement composition. Table 6-10 shows comparisons between heat of hydration and non-evaporable water and between the vapor sorption at 0.36 relative vapor pressure and non-evaporable water for the various types of cement. The results indicate that the type III cements evolve more heat per unit of non-evaporable water than do the other types of cement, whereas the sorption of hydrated type III cements per unit non-evaporable water is less than that of the other types of cement, type IV cements showing the greatest sorption.

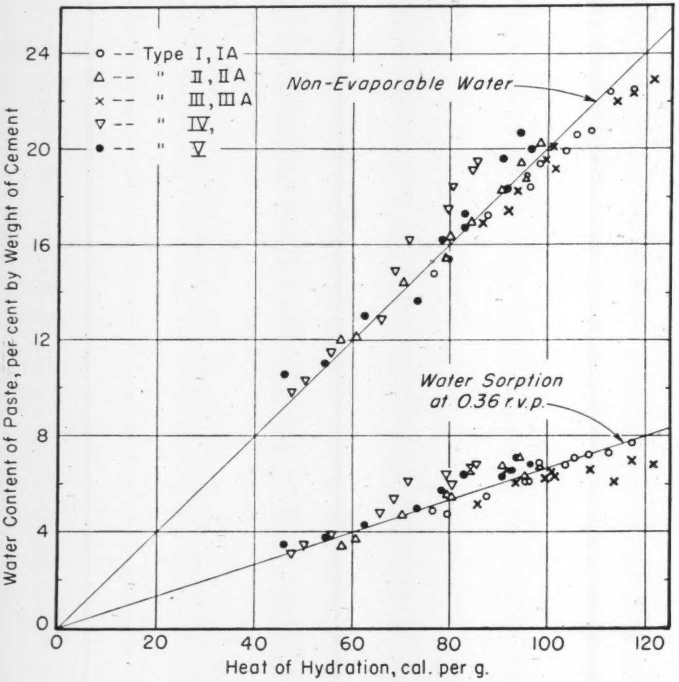


FIG. 6-7.—Relationship Between Heat of Hydration and Either Non-Evaporable Water or Water Sorption at 0.36 Relative Vapor Pressure.

Average results for each of five types of portland cement, pastes cured at 70 F. and mass cured, ages from 3 days to 6½ yr.

SUMMARY AND CONCLUSIONS

It may be stated on the basis of these tests of neat cement pastes having a water-cement ratio of 0.40 by weight that at this water-cement ratio:

1. The heat of hydration of portland cement increases continuously for at least 6½ yr.

2. The composition of the cement affects the heat of hydration at all ages. The heats of hydration of types II, IV, and V cements were consistently below those of the types I and III cements at all ages up to 6½ yr. Between 1 and 6½ yr. the rate of heat liberation of the types II, IV, and V cements was less than that of the types I and III cements.

3. The use of flake Vinsol resin as an air-entraining agent had no significant effect on the heat of hydration.

4. The results of least squares analyses indicate that somewhat better correlation is obtained between the heats of hydration and the calculated phase com-

positions of the cements than between the heats of hydration and the microscopically observed phase compositions.

5. Temperature of curing appreciably affects the rate of heat liberation at early ages. However, this effect does not persist, as companion pastes cured at 70 F. and 100 F. had approximately the same heats of hydration at 28 days.

6. The heat of hydration of cements at ages beyond 28 days cannot be estimated from strength data as other factors are involved in the relationship.

7. General relationships were observed between heat of hydration and either the non-evaporable water content or the vapor sorption of the pastes, although cement composition exerts some influence on these relationships.

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APPENDIX

TABLE 6-12.—EFFECT OF TEMPERATURE ON THE HEAT OF HYDRATION BY THE CONDUCTION CALORIMETER METHOD, UP TO 72 HOURS (6)

Neat pastes: water-cement ratio = 0.40 per cent by weight.

This table is a reproduction of Table 3-13, Chapter 3.

Temperature of Air and Bath, deg. Fahr.	Heat of Hydration, cal. per g. for Period Indicated									
	5 min.	10 min.	30 min.	1 hr.	4 hr.	8 hr.	12 hr.	24 hr.	48 hr.	72 hr.
CEMENT No. 11										
40	2.4	2.9	3.5	3.9	5.1	6.9	9.2	18.1	30.7	36.2
75	3.2	3.5	4.0	4.7	10.4	26.7	32.1	43.6	59.9	70.9
90	3.0	3.4	3.9	4.6	18.7	34.0	42.2	58.6	73.7	80.9
105	3.2	3.6	4.1	5.1	27.9	44.6	53.2	65.1	75.3	79.6
CEMENT No. 11T										
40	3.0	3.5	4.2	4.6	5.8	7.5	9.6	18.4	31.4	38.6
75	4.1	4.4	4.9	5.5	9.4	22.8	28.7	40.0	59.4	71.8
90	3.3	3.6	3.9	4.5	16.4	29.5	37.7	56.5	74.2	81.9
105	3.7	4.0	4.5	5.4	24.5	47.2	58.4	71.2	80.7	84.5
CEMENT No. 12										
40	2.5	3.0	4.2	4.7	7.1	8.9	10.5	16.3	27.5	33.7
75	3.8	4.4	4.9	5.6	9.0	20.7	26.9	39.6	55.5	68.3
90	4.1	4.6	5.0	5.5	11.9	27.3	34.0	47.6	64.4	73.0
105	4.2	4.8	5.1	5.9	19.2	36.1	46.1	61.8	75.9	83.1
CEMENT No. 12T										
40	2.4	2.8	3.8	4.3	6.6	8.4	9.8	15.6	26.7	33.7
75	3.8	4.1	4.9	5.6	9.1	20.2	26.7	38.0	52.7	64.0
90	3.8	4.3	4.9	5.6	11.8	26.6	34.0	49.3	66.8	76.7
105	4.0	4.5	5.2	6.0	19.4	36.0	45.6	61.3	76.5	83.8
CEMENT No. 13										
40	1.6	2.1	3.0	3.4	4.9	7.2	9.9	18.4	30.2	37.9
75	2.5	2.8	3.4	4.1	12.8	24.5	30.6	40.1	49.0	56.2
90	2.3	2.5	2.9	4.0	18.4	29.7	34.1	42.0	51.3	58.7
105	2.4	2.6	3.4	5.1	27.2	34.3	38.3	46.3	55.8	61.9
CEMENT No. 14										
40	2.7	3.5	4.3	4.9	6.9	9.9	12.6	21.3	35.0	43.0
75	4.4	4.8	5.4	6.5	14.6	27.7	35.3	48.7	64.5	72.8
90	4.8	5.1	5.5	6.5	22.2	36.4	43.9	57.8	67.8	72.6
105	4.7	5.0	5.5	6.6	28.0	45.1	51.4	59.4	65.2	69.2
CEMENT No. 15										
40	2.2	2.8	3.7	4.1	6.0	8.1	10.2	19.8	34.6	40.9
75	3.0	3.3	3.7	4.0	8.9	25.0	34.2	42.7	54.2	62.6
90	3.5	3.7	4.0	4.5	15.8	46.0	52.7	64.5	75.5	81.7
105	3.7	3.9	4.2	4.9	32.5	47.5	54.9	66.9	78.5	87.4
CEMENT No. 16										
40	2.0	2.7	3.8	4.2	5.9	8.2	10.8	20.3	32.7	38.2
75	3.0	3.3	3.7	4.0	8.9	25.0	34.2	42.7	54.2	62.6
90	2.7	3.0	3.1	3.8	13.5	32.9	38.0	47.4	59.5	66.8
105	2.8	3.1	3.5	4.1	28.2	42.9	49.1	60.1	82.4	88.6
CEMENT No. 16T										
40	1.9	2.5	3.5	3.9	5.7	8.2	10.7	18.5	31.5	38.1
75	3.1	3.4	3.8	4.1	7.7	23.0	34.5	43.9	55.5	64.1
90	3.0	3.1	3.1	3.4	11.0	34.1	40.0	48.8	61.4	69.2
105	3.2	3.4	3.7	4.3	25.9	44.5	51.2	62.7	75.8	83.1
CEMENT No. 17										
40	2.6	3.4	4.1	4.7	7.7	10.7	13.1	20.5	29.6	34.7
75	3.7	4.0	4.4	5.1	11.5	27.4	33.3	43.4	57.5	66.4
90	3.6	3.8	3.8	4.6	15.8	33.7	40.4	52.1	65.8	72.8
105	3.7	3.9	4.3	5.2	27.8	41.2	48.1	59.4	72.3	81.9

TABLE 6-12.—*Continued.*

Temperature of Air and Bath, deg. Fahr.	Heat of Hydration, cal. per g. for Period Indicated									
	5 min.	10 min.	30 min.	1 hr.	4 hr.	8 hr.	12 hr.	24 hr.	48 hr.	72 hr.
					Cement No. 18					
40	2.2	2.7	3.4	3.8	4.9	6.4	8.2	16.2	28.9	33.9
75	3.4	3.8	4.4	5.1	9.6	26.1	34.1	45.8	59.7	70.0
90	3.3	3.9	4.7	5.5	15.2	32.8	39.8	52.6	67.0	75.9
105	3.0	3.2	3.8	5.0	26.7	40.8	47.5	58.7	70.8	79.0
					Cement No. 18T					
40	2.1	2.8	3.7	4.4	5.9	8.0	10.4	18.9	26.9	33.6
75	4.5	5.1	5.6	6.5	10.6	20.2	24.5	41.5	60.4	72.8
90	4.8	5.2	5.9	6.8	17.0	24.1	33.7	50.8	66.8	76.5
105	4.0	4.7	5.2	11.7	18.1	34.6	44.5	57.8	70.4	78.0
					Cement No. 21					
40	1.5	2.3	3.2	3.5	5.5	7.1	8.1	12.4	19.9	23.5
75	2.9	3.2	3.6	4.1	7.6	18.0	23.8	31.7	43.5	50.8
90	2.7	2.8	2.8	3.1	9.6	22.1	27.6	37.8	49.1	54.2
105	2.9	3.1	3.5	3.8	15.8	26.6	33.2	43.9	55.3	62.5
					Cement No. 21T					
40	1.7	2.3	3.6	3.8	5.8	6.4	7.0	10.6	16.1	18.2
75	3.1	3.3	3.7	4.3	7.7	17.7	22.4	31.3	44.3	52.2
90	3.1	3.2	3.3	3.7	9.9	21.0	26.5	37.4	49.2	55.3
105	3.3	3.6	3.8	4.2	15.2	26.9	34.8	47.1	59.6	66.6
					Cement No. 22					
40	1.9	2.7	3.7	4.4	7.0	8.1	9.3	14.2	23.3	27.9
75	3.5	4.0	4.6	5.4	8.3	17.5	23.2	30.7	41.6	50.0
90	3.5	3.8	4.1	4.8	9.8	22.4	27.2	36.0	50.6	58.8
105	3.4	3.9	4.4	4.9	15.3	29.1	36.7	49.2	61.0	67.4
					Cement No. 23					
40	1.7	2.3	3.1	3.5	5.3	6.8	8.3	13.3	23.9	29.9
75	3.0	3.4	3.9	4.5	8.7	18.4	25.5	35.8	44.8	51.3
90	2.6	2.9	3.2	3.7	11.7	25.1	31.7	38.7	46.9	52.0
105	2.6	2.8	3.1	3.3	15.2	27.7	32.2	42.2	52.7	60.8
					Cement No. 24					
40	1.7	2.7	3.8	4.4	5.6	7.9	10.6	19.1	31.2	35.5
75	3.2	3.6	4.1	4.8	12.0	28.0	33.1	41.8	54.2	62.4
90	3.4	3.7	4.0	4.7	20.9	31.9	37.7	48.1	59.4	64.0
105	3.8	4.2	4.6	5.7	26.7	37.6	43.8	53.6	60.7	65.0
					Cement No. 25					
40	1.8	3.0	4.2	5.0	7.3	10.3	12.7	19.5	31.0	36.5
75	3.6	3.9	4.1	4.6	8.8	21.6	29.1	37.1	47.5	54.9
90	3.6	3.7	3.9	4.3	12.8	29.1	33.2	41.6	51.8	58.6
105	3.6	3.7	4.0	4.4	21.9	35.4	41.7	50.6	59.5	63.6
					Cement No. 31					
40	3.5	4.3	5.0	5.9	10.3	14.9	18.1	31.7	45.8	54.6
75	5.6	6.1	6.7	8.2	25.8	43.1	47.4	58.0	74.7	83.7
90	5.5	5.8	6.5	8.5	37.6	49.9	56.1	71.0	84.8	90.7
105	5.7	6.2	7.2	10.4	45.5	60.1	68.0	81.5	91.5	97.3
					Cement No. 33					
40	2.3	3.2	4.6	5.2	8.8	13.7	17.6	28.4	42.0	49.1
75	4.4	4.8	5.3	6.4	22.5	44.5	52.1	63.3	77.5	86.3
90	4.5	4.8	5.4	6.9	32.8	51.5	57.6	68.7	80.6	85.4
105	5.0	5.3	5.8	7.0	46.8	61.9	69.5	81.6	89.7	95.2

(Continued on page 1260.)

TABLE 6-12.—*Concluded.*

Temperature of Air and Bath, deg. Fahr.	Heat of Hydration, cal. per g. for Period Indicated									
	5 min.	10 min.	30 min.	1 hr.	4 hr.	8 hr.	12 hr.	24 hr.	48 hr.	72 hr.
CEMENT NO. 33T										
40	2.8	3.4	4.5	5.1	8.6	13.2	17.0	28.3	44.4	53.6
75	4.5	4.9	5.5	6.7	24.0	48.1	54.9	66.7	81.2	87.9
90	4.4	4.7	5.3	7.0	33.4	52.1	58.1	71.0	84.3	89.1
105	4.9	5.2	5.6	7.7	46.9	62.4	70.1	80.7	88.8	94.6
CEMENT NO. 34										
40	1.8	2.6	4.3	5.3	8.6	12.0	15.4	26.5	44.3	54.1
75	4.4	5.0	5.7	6.7	19.8	39.4	45.4	54.5	67.0	75.1
90	4.4	4.7	4.9	6.1	30.6	44.3	49.5	60.5	71.8	75.9
105	4.8	5.0	5.3	6.3	34.8	47.6	55.0	68.7	79.0	85.8
CEMENT NO. 41										
40	1.9	2.7	3.6	4.4	5.8	8.6	10.9	16.5	23.9	26.6
75	3.4	3.8	4.4	5.0	9.1	20.7	26.2	33.1	43.7	50.4
90	3.3	3.5	3.6	4.0	13.5	25.5	29.0	37.5	44.0	46.8
105	4.1	4.3	4.5	5.1	20.7	29.4	34.3	42.5	48.3	51.8
CEMENT NO. 42										
40	1.0	1.9	2.9	3.3	4.4	6.3	8.3	13.1	20.3	24.5
75	2.1	2.6	3.1	3.4	5.1	12.9	21.2	30.6	37.2	41.6
90	2.0	2.2	2.3	2.5	5.5	18.2	24.5	30.4	36.8	40.8
105	2.7	2.8	3.2	3.3	7.9	23.5	27.4	34.4	41.4	46.3
CEMENT NO. 43										
40	3.0	4.1	5.2	5.8	8.6	10.2	11.7	15.6	23.5	27.3
75	5.0	5.3	5.6	6.1	10.6	20.3	24.5	31.2	43.3	51.0
90	4.8	5.1	5.1	5.7	15.5	25.4	29.6	42.8	50.4	54.1
105	5.0	5.3	5.4	6.1	22.1	32.4	39.7	47.2	52.6	55.8
CEMENT NO. 43A										
40	2.1	2.9	4.0	4.6	7.8	9.7	11.6	16.0	21.4	24.4
75	3.4	3.7	4.1	4.9	11.3	20.4	26.0	32.7	38.9	43.2
90	3.1	3.2	3.1	4.0	14.8	24.8	28.9	34.4	38.5	41.5
105	3.3	3.5	3.6	4.8	20.7	28.4	31.7	37.9	45.0	51.0
CEMENT NO. 51										
40	1.0	1.7	3.0	3.6	6.0	8.0	10.0	15.9	25.9	31.5
75	2.8	3.2	3.7	4.1	8.7	20.6	27.7	37.6	45.9	50.8
90	2.3	2.4	2.4	2.9	10.1	23.3	29.4	37.7	46.0	51.4
105	2.5	2.7	2.9	3.3	15.1	29.2	34.3	43.0	53.6	61.5

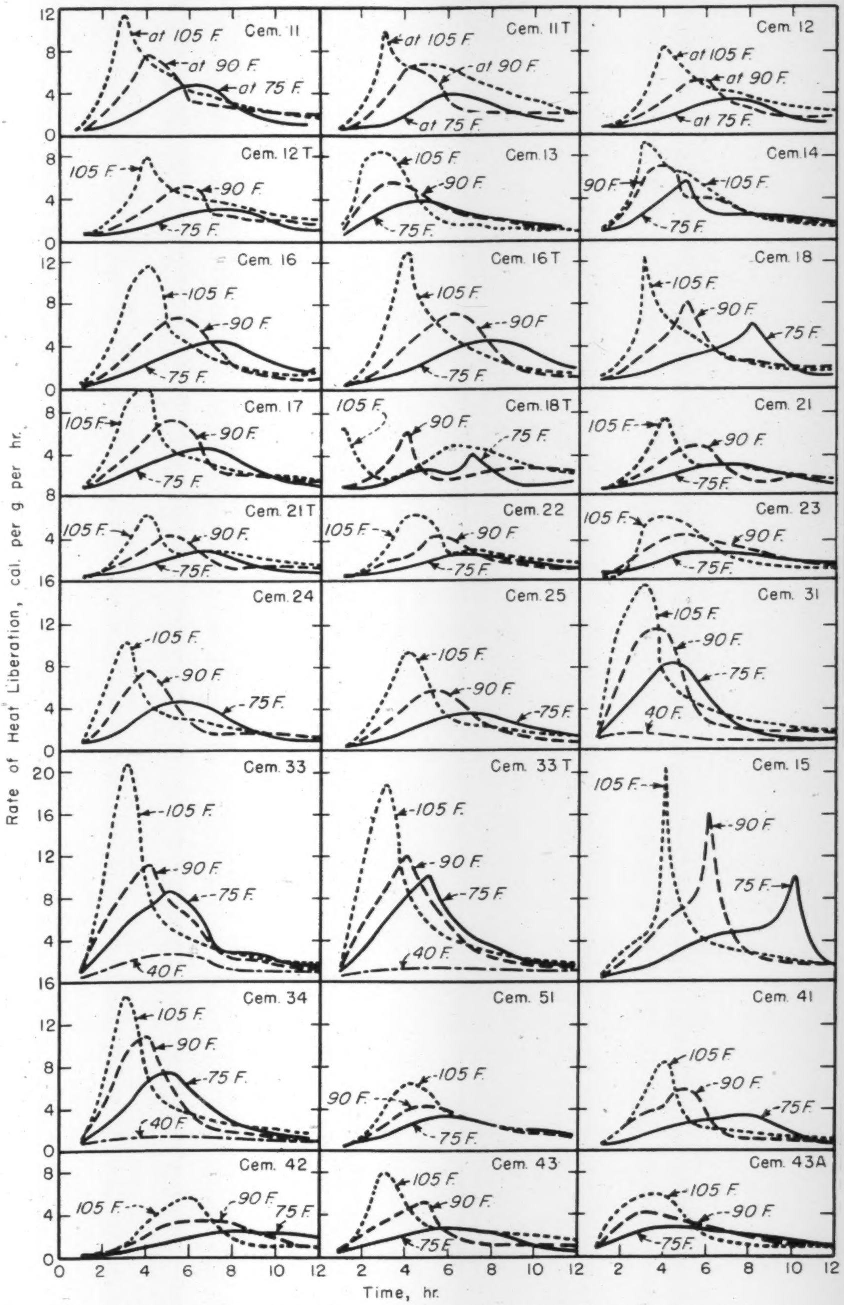
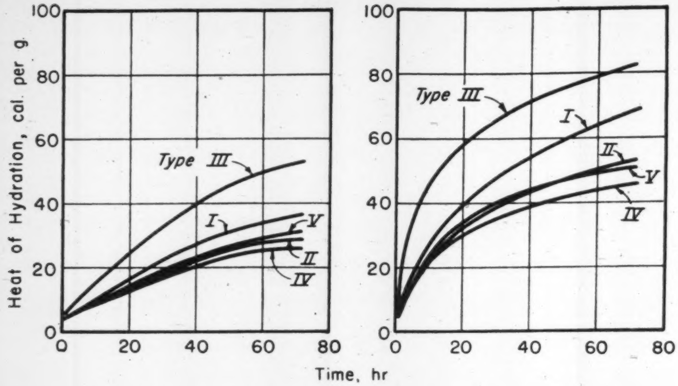
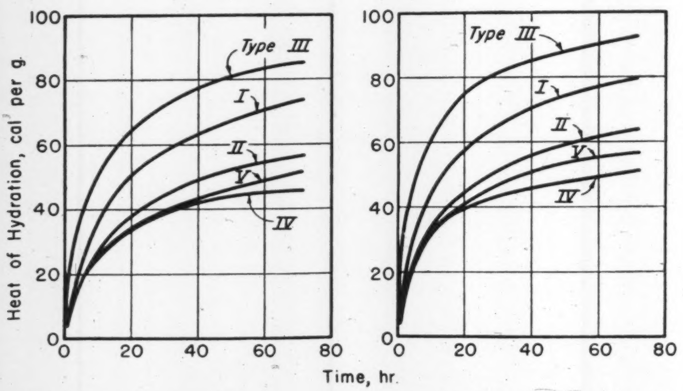


Fig. 6-8.—Rates of Heat Liberation at Three Temperatures.
This figure is a reproduction of Fig. 3-1, Chapter 3 (6).



(*a*) Heat of hydration at 40 F.

(*b*) Heat of hydration at 75 F.



(*c*) Heat of hydration at 90 F.

(*d*) Heat of hydration at 105 F.

Fig. 6-9.—Average Heat of Hydration of the Different Types of Portland Cement—Conduction Calorimeter, Data taken from Table 6-13.

This figure is a reproduction of Fig 3-2, Chapter 3 (6).

TABLE 6-13.—AVERAGE HEATS OF HYDRATION OF THE DIFFERENT TYPES OF PORTLAND CEMENTS AT EARLY AGES.

Cement Type	Number Averaged	Temperature of Air and Bath, deg. Fahr.	Heat of Hydration at Age Indicated, cal. per g.									
			5 min.	10 min.	30 min.	1 hr.	4 hr.	8 hr.	12 hr.	24 hr.	48 hr.	72 hr.
I and IA	12	40	2.3	2.9	3.8	4.2	6.0	8.2	10.4	18.5	30.5	36.9
II and IIA	6	40	1.7	2.6	3.6	4.1	6.1	7.8	9.3	14.9	24.2	28.6
III and IIIA	4	40	2.6	3.4	4.6	5.4	9.1	13.5	17.0	28.7	44.1	52.9
IV	4	40	2.0	2.9	3.9	4.5	6.7	8.7	10.6	15.3	22.3	25.7
V	1	40	1.0	1.7	3.0	3.6	6.0	8.0	10.0	15.9	25.9	31.5
I and IA	12	75	3.5	3.9	4.2	5.1	10.2	24.1	32.2	43.7	58.2	68.0
II and IIA	6	75	3.2	3.6	4.0	4.6	8.8	20.2	26.1	34.7	45.9	53.6
III and IIIA	4	75	4.7	5.2	5.6	7.0	23.0	43.7	49.9	60.6	75.1	83.2
IV	4	75	3.5	3.9	4.3	4.9	9.0	18.8	24.5	31.9	40.8	46.6
V	1	75	2.8	3.2	3.7	4.1	8.7	20.6	27.7	37.6	45.9	50.8
I and IA	12	90	3.5	3.9	4.2	4.9	15.6	32.3	39.2	52.3	66.2	73.9
II and IIA	6	90	3.2	3.4	3.6	4.0	12.5	25.3	30.7	39.9	51.2	57.1
III and IIIA	4	90	4.7	5.0	5.5	7.1	33.6	49.5	55.3	67.8	80.4	85.3
IV	4	90	3.3	3.5	3.6	4.1	12.3	23.5	28.0	36.3	42.4	45.8
V	1	90	2.3	2.4	2.9	2.9	10.1	23.3	29.4	37.7	46.0	51.4
I and IA	12	105	3.5	3.9	4.4	5.8	25.5	41.2	49.0	60.9	73.3	80.0
II and IIA	6	105	3.3	3.6	3.9	4.4	18.4	30.6	37.1	47.8	58.1	64.3
III and IIIA	4	105	5.1	5.4	5.9	7.9	43.5	58.0	65.7	78.1	87.3	93.2
IV	4	105	3.8	3.9	4.2	4.8	17.9	28.4	33.3	40.5	46.8	51.2
V	1	105	2.5	2.7	2.0	3.3	15.1	29.2	34.3	43.0	53.6	61.5

THE EFFECT OF TIME AND TEMPERATURE OF BURNING ON THE PROPERTIES OF QUICKLIME PREPARED FROM CALCITE*

By J. A. Murray,[1] H. C. Fischer,[1] and D. W. Sabean[1, 2]

Synopsis

Pure calcite, sized to pass a No. 10 sieve and to be retained on a No. 20 sieve, was calcined in an electrically heated rotary tube, which was provided with continuous feed and discharge. Over 40 calcinations were made, with maximum material temperatures varying from 1700 F. to 2400 F. and with over-all retention times varying from 46 min. to 474 min. The density, porosity, surface area, and reactivity with water were determined on each of the resulting quicklimes.

Porosity, surface area, and reactivity with water are closely related. Highly porous limes have high surface area and a fast rate of reaction, while limes of low porosity are characterized by low surface area and slow reaction with water.

The results show that porosity of the quicklime is determined primarily by the rate of heating prior to and during calcination, while the heating conditions subsequent to calcination have relatively little effect. When material of this size required 35 min. or more to reach a temperature of 1700 F., the resulting quicklime was always highly porous, regardless of subsequent treatment. Quicklimes calcined in increasingly shorter periods were correspondingly less porous.

The properties of quicklime and of the products made from quicklime are affected by many factors such as the composition and texture of the original limestone, the methods used in burning the stone, and the handling and exposure conditions to which the lime is subject during cooling and storage. The present report is concerned only with the effect of burning conditions upon the activity, porosity, and surface area of quicklimes produced from a pure crystalline calcite. Some of the other variables are still under investigation and will be the subject of later reports.

Commercially, quicklime is produced in both rotary and shaft kilns. The products of the two types of kilns differ markedly, even when made from the same stone. This is first shown by the variation in activity of the two types of lime upon treating with water, and subsequently by differences in the hydrated products; putties and hydrates produced from the two types of lime have different settling rates, yields, and flow characteristics.

Many of these differences have been attributed to the wide difference in retention time in the kiln. It is not unusual for material to be in a shaft kiln for 75 to 100 hr., whereas under rotary kiln operation the time from cold stone to cold quicklime may be 2 hr. or less. The time ratio of about 50:1 may well be expected to produce a difference in properties.

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The temperatures which prevail in the two types of kilns are not well known. In a rotary kiln, it is difficult to measure anything but the momentary surface temperatures of the particles as they slide down the bed of tumbling material. In a shaft kiln, it is impossible to see or measure anything but the lumps at the edge of the kiln. The results are that the maximum temperatures in commercial kilns are largely unknown, but the opinions of operating men are that they probably lie in the range of 1800 to 2300 F. in both rotary and shaft kilns.

One of the earliest studies of the effect of temperature of burning on the properties of quicklimes was that by Bleininger and Emley (**1**)[3] They burned 4-lb. samples of stone, encased in saggers, in a downdraft muffle kiln at a series of controlled temperatures. They found that the slaking reaction was a means of fixing the proper burning temperature for a given lime and that the relation between porosity and calcining temperature gave valuable information in regard to the best calcining temperature.

The work of Bleininger and Emley was done under static conditions with no variations being made in the heating schedule. Other investigators (**2**, **3**, **4**) have similarly burned commercial stones under static conditions. No references have been found to burning under controlled conditions of agitation on a laboratory scale.

One of the objections to burning under static conditions is the difficulty of obtaining an accurate determination of the variation of temperature of the stone with time. If stone is to be burned in a muffle, the muffle may initially be at the desired temperature but the introduction of the cold stone drops the muffle temperature. A later recovery takes place, but it is impossible to determine exactly when and how closely the charge approaches the muffle temperature.

Another objection to burning under static conditions is that it usually requires the charge to be in lumps of $\frac{1}{4}$-in. or larger, a size too large for laboratory tests. Any necessary grinding or pulverizing, particularly in a humid atmosphere and with an active lime, results in an uncontrolled pickup of carbon dioxide and water and produces a change of unknown magnitude in the activity and possibly in other properties of the lime.

Burning in a rotary kiln therefore appears to have several advantages over static burning for laboratory investigations. It permits accurate control and measurement of the time-temperature relations, it utilizes stone of small size so that the lime produced may be evaluated without further grinding and therefore with minimum opportunity for accidental pickup of carbon dioxide and water, and finally, the rotary kiln permits control of the atmosphere during burning. For these reasons, a rotary kiln was selected as the preferable calcining unit for this study.

Impurities in limestones may have an important effect upon the properties of the resulting quicklimes. Since later portions of the program will be concerned with the evaluation of the effect of impurities, it is desirable to have data obtained on a stone without impurities for comparison. Consequently, a crystalline calcite was used as a raw material for these tests.

The properties of the quicklimes which

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1277.

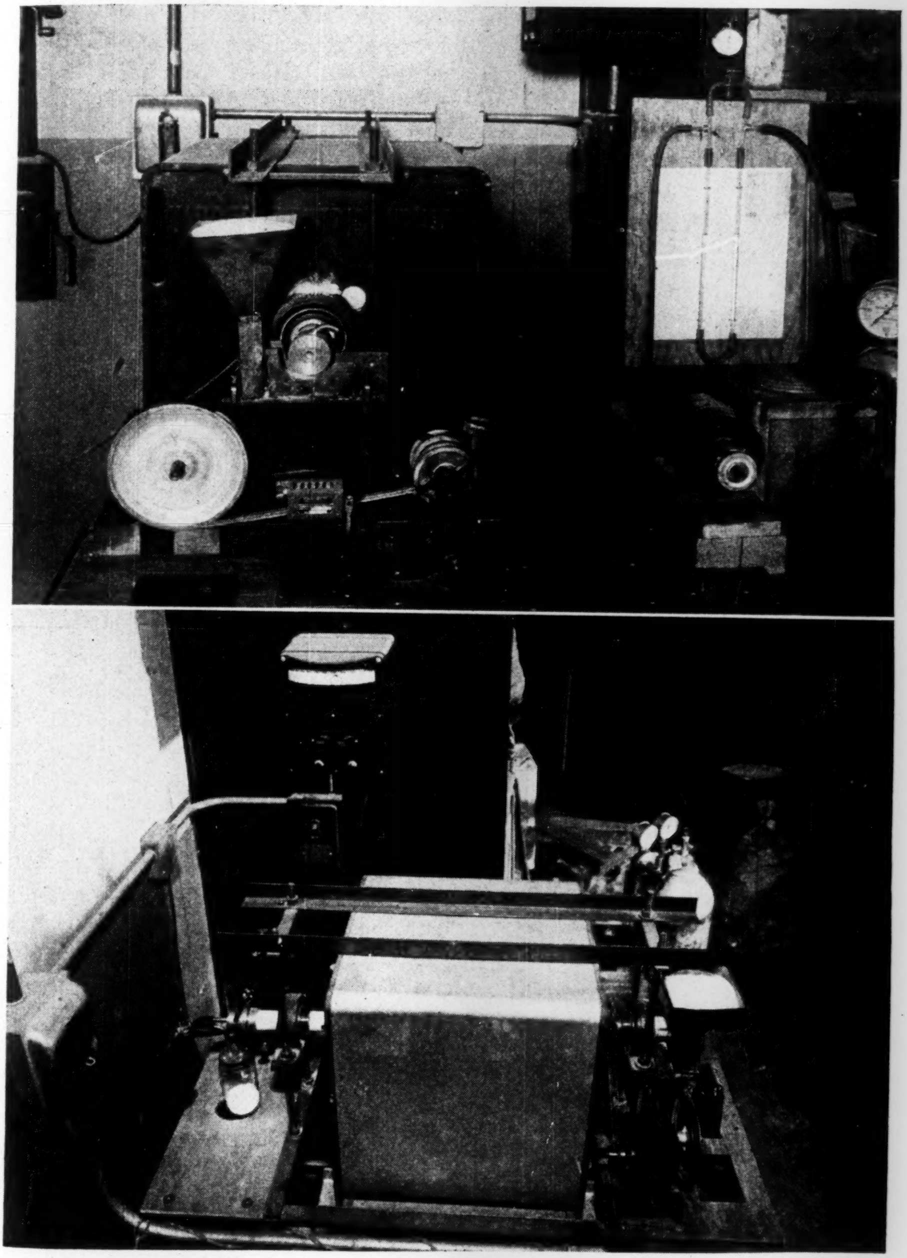

Fig. 1.—Rotary Kiln—Feed End and Side View.

were selected for study were reactivity with water, porosity, and surface area. Reactivity with water is the one of most interest from the user's viewpoint. All three of these properties are more or less interrelated, for a high degree of activity is usually associated with a highly porous lime which also has high surface area.

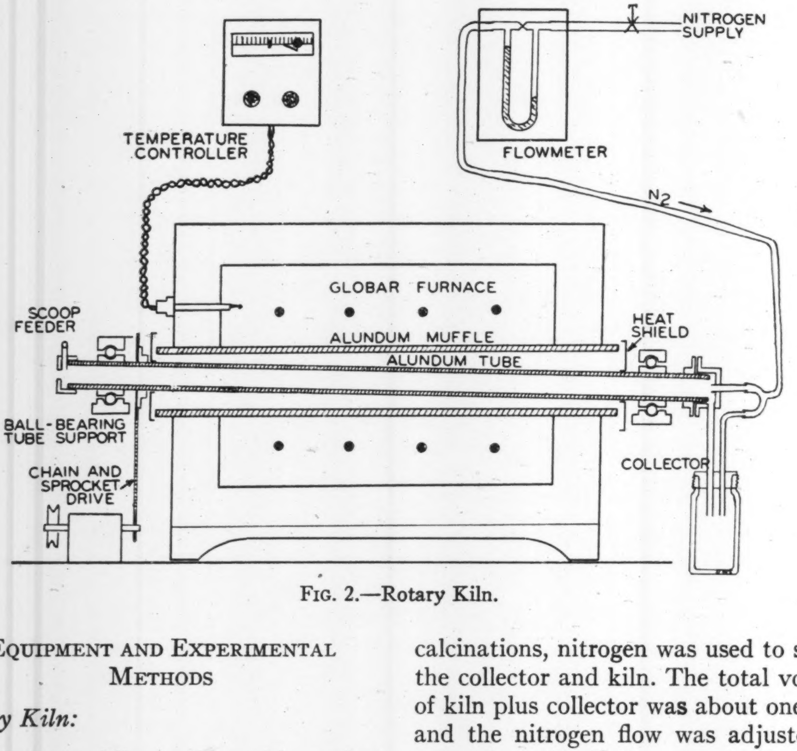

FIG. 2.—Rotary Kiln.

EQUIPMENT AND EXPERIMENTAL METHODS

Rotary Kiln:

The rotary kiln used on this project consisted of an alundum tube, $1\frac{1}{8}$ in. inside diameter and 36 in. long, mounted within an electrically heated muffle furnace which was provided with automatic temperature control. The kiln was mounted with a pitch of $\frac{1}{4}$ in. per foot and was equipped with a variable speed drive so that the rotation could be controlled at will within the range of 0.78 to 8.0 rpm. A scoop-type feeder, which picked up a uniform amount of the calcite on each revolution of the kiln, was used. The discharge from the kiln was collected directly in a glass screw-top bottle so that the transfer from one container to another would be avoided. An attachment held the collector on the discharge end of the kiln in such a manner that the collector and kiln could be purged continously with an inert gas. For all calcinations, nitrogen was used to sweep the collector and kiln. The total volume of kiln plus collector was about one liter and the nitrogen flow was adjusted to approximately $1\frac{1}{4}$ l. per min. A small opening was left at the feed end of the kiln so that a probe thermocouple could be inserted, allowing temperature measurements to be made at any desired point over the length of the kiln. The end of the thermocouple was bent at an angle so that the bead of the couple could be inserted and held in the tumbling bed of material. This permitted the measurement of the temperature distribution in the material in the kiln. The muffle furnace which enclosed the kiln was automatically controlled to ±20 F. by a control mechanism actuated by a thermocouple located in the muffle, above the kiln. The temperature fluctuations in the kiln were of appreciably less magnitude than those in the muffle. Figure 1 shows the feed end and side view of the kiln. Figure 2 is a line drawing showing the essential details of the construction.

Calcination in a rotary kiln normally causes some size reduction in the stone due to abrasion and thermal shock. The resulting quicklime may undergo an additional size reduction due to abrasion and also by thermal shrinkage. However the calcite used in this study produced a lime, the gradation of which was substantially that of the calcite feed. Little if any shrinkage with practically no dusting occurred during the change from carbonate to oxide.

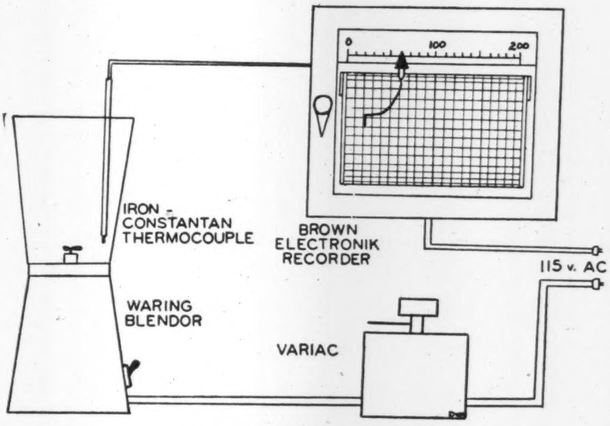

FIG. 3.—Slaking Apparatus.

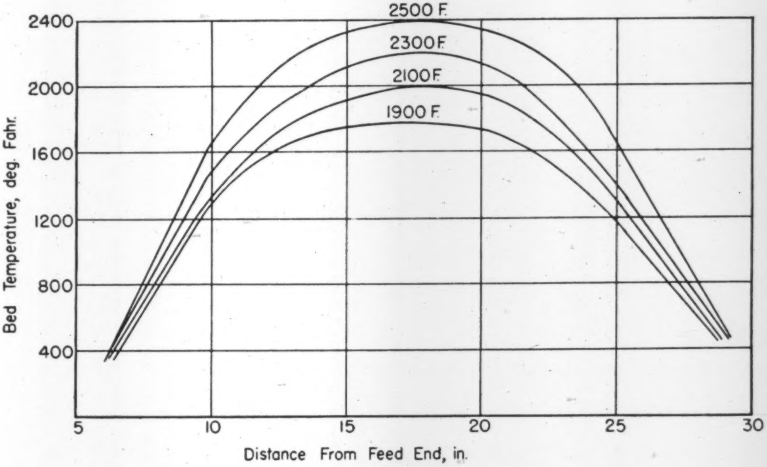

FIG. 4.—Typical Temperature Distribution in Kiln at Various Muffle Temperatures.

Lime Slaker:

To establish the reactivity of the lime with water, it was felt that a measurement of the temperature rise on slaking would be a suitable index. It was obvious that, because of the extreme rapidity with which some of these limes slaked, it would be impossible to follow the temperature rise with a mercury thermometer; consequently, an iron-constantan thermocouple was used with a Brown Electronik Recorder. The speed of the recorder was such that the pen will make a complete sweep across the 11-in. chart (0 to 200 C.) in about 2 sec. Since a water-lime ratio was selected which would give a maximum temperature rise of about 40 C. or one-fifth of the chart width, the maximum instrument lag would be less than one-half second, if the reaction were instantaneous. The recorder chart was graduated in 1 C. intervals and could be read to 0.1 C. With a chart speed of 2 in. per min., the time scale could be read to 1 sec.

TABLE I.—PROPERTIES OF QUICKLIMES AS AFFECTED BY TEMPERATURE AND TIME OF CALCINATION.

Calcination	Maximum Temperature of Bed, deg. Fahr.	Retention Time, min.	Time to Reach 1700 F., min.	Ignition Loss, per cent	CO_2, per cent	Surface Area, sq. meters per g.	Porosity, per cent	Bulk Specific Gravity	Activity Coefficient, ΔT_5
19-1	1850	474	182	1.7	0.2	11.3	46.6	1.78	33.4
19-2	1850	230	88	0.8	0.1	13.8	47.8	1.74	35.2
19-3	1834	185	72	...	0.1	16.1	48.1	1.73	35.2
19-4	1857	103	39	0.1	0.1	16.1	48.1	1.73	36.3
19-5	1850	97	37	0.2	0.1	16.6	48.1	1.73	36.3
19-6	1760	82	34	0.3	0.1	16.2	48.4	1.72	35.5
19-7	1727	74	31	...	0.3	16.2	48.4	1.72	36.3
19-8	1707	67	29	2.9	2.1	17.1	48.4	1.72	33.2
21-1	2020	474	163	1.9	0.1	8.15	46.0	1.80	30.2
21-2	2018	230	79	0.6	0.1	13.1	47.2	1.76	34.3
21-3	2020	103	35	...	0.1	12.3	46.6	1.78	33.6
21-4	2023	95	33	...	0.1	12.2	43.9	1.87	30.5
21-5	2023	92	32	0.1	0.1	9.55	44.2	1.86	30.9
21-6	2020	82	28	0.2	0.1	11.8	43.3	1.89	26.0
21-7	2000	76	27	...	0.1	11.5	43.9	1.87	26.6
21-8	2000	67	23	0.1	0.1	11.2	42.4	1.92	22.3
21-9	1853	63	24	...	0.4	7.49	37.0	2.10	11.4
21-10	1780	49	20	1.2	1.1	9.50	41.2	1.96	14.2
21-11	1765	46	19	1.1	1.1	8.80	38.4	2.05	12.0
23-1	2218	474	146	1.1	0.1	8.40	45.7	1.81	31.5
23-2	2220	474	146	1.1	0.1	9.82	46.3	1.79	33.2
23-3	2223	308	95	1.1	0.1	10.9	46.3	1.79	33.8
23-4	...	230	71	0.9	0.1	9.50	45.7	1.81	31.9
23-5	2229	127	39	0.1	0.1	7.60	33.7	2.21	7.9
23-6	2225	103	36	0.1	0.1	7.45	34.0	2.20	8.4
23-7	2218	82	25	0.1	0.1	7.48	34.9	2.17	11.4
23-8	2204	67	21	0.1	0.1	6.40	31.0	2.30	6.4
23-9	...	66	20	0.1	0.1	5.60	29.7	2.34	6.7
23-10	2158	48	15	0.1	0.1	6.59	29.4	2.35	5.1
23-11	2184	47	15	0.7	0.6	4.54	25.8	2.47	5.5
25-1	2362	474	136	0.3	0.1	4.28	43.5	1.88	20.8
25-2	2397	308	86	0.8	0.1	7.62	45.0	1.83	30.2
25-3	2400	230	65	0.6	0.1	8.60	44.5	1.85	28.7
25-4	2396	103	29	0.2	0.1	4.17	27.9	2.40	7.2
25-5	...	100	28	0.1	0.1	3.26	33.6	2.21	9.6
25-6	2412	95	26	...	0.1	2.64	21.6	2.61	3.6
25-7	2402	82	23	0.1	0.1	2.91	21.6	2.61	2.2
25-8	2397	74	21	...	0.1	1.81	18.6	2.71	2.0
25-9	2407	67	19	0.1	0.1	2.28	20.4	2.65	2.6
25-10	2402	49	14	0.2	0.1	2.53	21.0	2.63	2.0
25-11	2341	48	14	0.2	0.1	2.35	19.8	2.67	2.4

A Waring Blendor was used for slaking the lime. This gave such violent agitation that it was necessary to decrease the speed by reducing the voltage. Figure 3 is a line drawing of the slaking apparatus. The entire equipment was operated in a constant temperature room, controlled at 20 C. The quicklimes and the distilled water for slaking were both stored in closed containers in the constant temperature room overnight, before the slaking test was made. A ratio of 7 parts of water to 1 part of quicklime by weight was used for all slaking tests. This ratio provided enough water to prevent loss of heat in evolving steam, but not enough to cause drowning.

Surface Area Equipment and Measurements:

The surface areas of the quicklimes were determined by the low-temperature nitrogen adsorption method, based on the Brunauer, Emmett, and Teller Theory (5). The apparatus and techniques used were the same as reported by Staley and Greenfeld (6) in evaluating quicklimes and hydrates produced from a commercial limestone.

Density and Porosity:

The true densities of these materials were determined by helium displacement and the apparent densities by mercury displacement. From these two densities the porosity was calculated by the following formula:

$$P = \left(\frac{D_{He} - D_{Hg}}{D_{He}}\right) \cdot 100$$

where:

P = porosity in per cent,
D_{He} = density by helium displacement, and
D_{Hg} = density by mercury displacement.

Loss on Ignition and Carbon Dioxide Content of Lime:

The values for loss on ignition and for carbon dioxide content were used to establish the completeness of burning and as an index of the amount of air slaking which had occurred during handling. The material was sampled after cooling overnight in a closed container. The methods used for these determinations were those of A.S.T.M. Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (C 25 - 44).[4] The results are given in Table I to the nearest 0.1 per cent, except in the case of several CO_2 determinations which were in the range of 0.02 to 0.04 per cent and which are reported as 0.1 per cent.

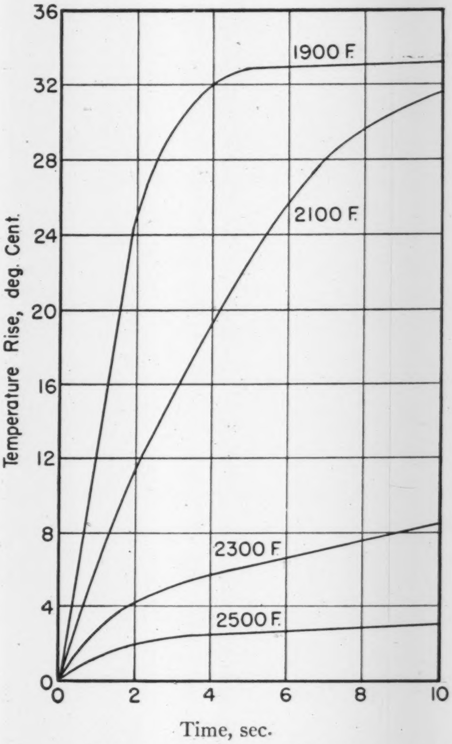

FIG. 5.—Effect of Calcining Temperature on Slaking Activity of Lime.
Retention Time = 67 min.
Numbers Represent Muffle Temperatures.

Calcite:

The calcite used in this investigation was the type designated as "cleavable calcite" by the supplier. As received it consisted of crystals ranging in size from $\frac{1}{4}$ in. up to 2 in. Most of the crystals were clear and colorless, but a few had some surface impurities. The crystals were hand-picked to eliminate any containing obvious impurities. The selected crystals were then crushed and screened to pass a No. 10 and to be retained on a No. 20 sieve. The sized material was washed with water to remove adhering dust of fracture and dried in an electric oven before use.

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[4] 1946 Book of A.S.T.M. Standards, Part II, p. 114.

The material thus prepared was of such quality that the impurities could not be detected by normal wet analytical methods. Its purity was confirmed by spectrographic analysis.

The density of the calcite as determined by both helium and mercury displacement was found to be 2.70 g. per cu. cm. Surface area of the calcite was determined as 0.015 sq. m. per g.

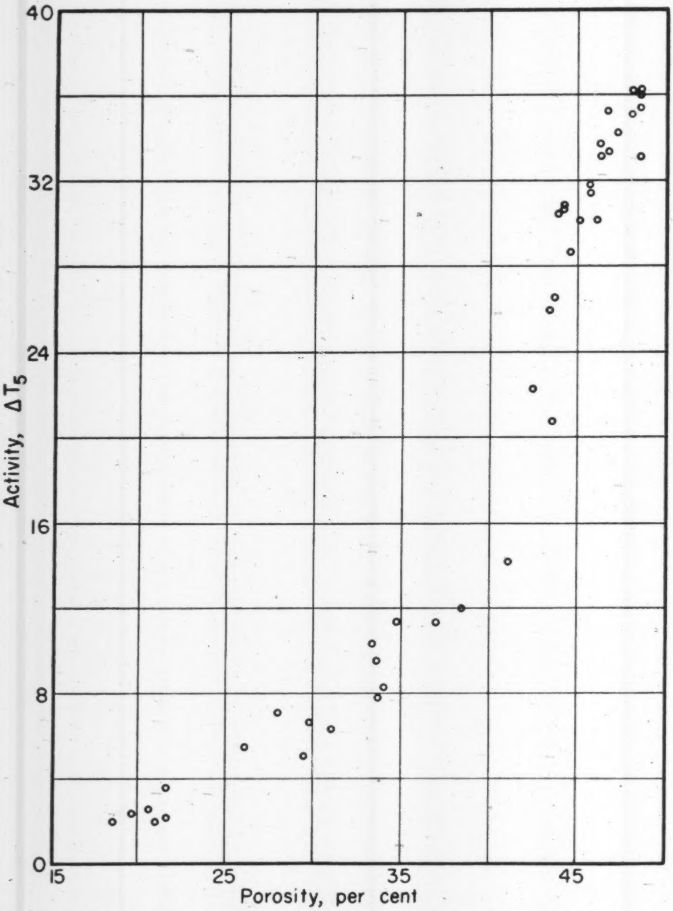

FIG. 6.—Relation Between Activity and Porosity of Lime.

RESULTS

Temperature Distribution in Kiln:

The temperature distribution in the kiln was measured by inserting the probe thermocouple through the feed end of the kiln. Measurements were made at 2-in. intervals with the thermocouple bead embedded in the charge. Typical kiln temperature distribution curves are shown in Fig. 4 for muffle temperatures from 1900 to 2500 F. In general, the point of maximum temperature was close to the center point of the kiln, and the maximum kiln temperature was roughly 100 F. less than the temperature at which the muffle was controlled. The maximum kiln temperature decreased as the retention time of the material in the kiln was decreased. In some cases the maximum temperature in the kiln was almost 200 F. lower than the indicated muffle temperature when the retention time was very low.

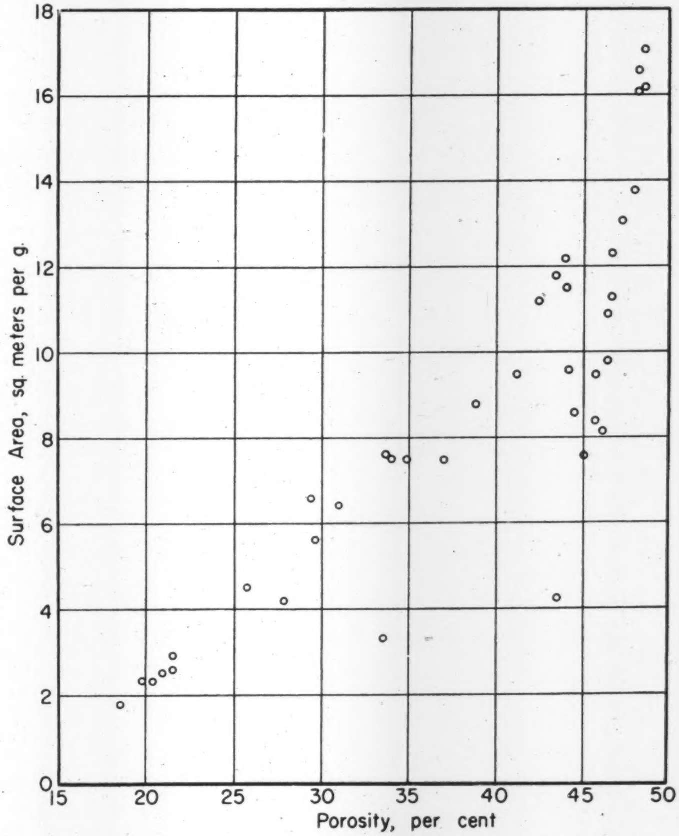

FIG. 7.—Relation Between Surface Area and Porosity of Lime. Temperatures Represent Muffle Temperatures.

Activity:

It is obviously desirable to have some single numerical value which will be indicative of the activity of the quicklime when slaked with water. However, it is extremely difficult to obtain such a figure which can be justified on theoretical grounds. Careful study of the slaking curves indicates that at least two types of curves were produced. When temperature rise is plotted against time using linear coordinates it is found that the curves representing the active limes are practically straight until the major portion of the quicklime has been exhausted. On the other hand, the inactive limes produce a curve which is hyperbolic in character. The plot of temperature rise against time is practically straight on log scales for a large part of the slaking period. A few limes appear to be intermediate in character between these extremes, and do not give straight-line plots on either linear, semi-log, or log plotting.

Because of the variation in type of slaking curve, an empirical point was selected to be indicative of the rapidity of slaking. The point selected was the temperature rise which had occurred 5 sec. after adding the quicklime to the water. The "activity" determined in this manner is designated by the symbol ΔT_5. This point is admittedly a compromise, but has proved to be very useful in comparing the several limes. The most serious error in using it as an index of activity is found with the extremely active limes. With some of these, the slaking was complete in 3 to 4 sec., and the reading at 5 sec. gives the impression that these limes are somewhat slower to slake than is actually the case. Figure 5 presents typical slaking curves and illustrates the effect of variation in kiln temperature on the rate of development of heat. The limes represented by the curves of Fig. 5 had a uniform retention time in the kiln of 67 min.

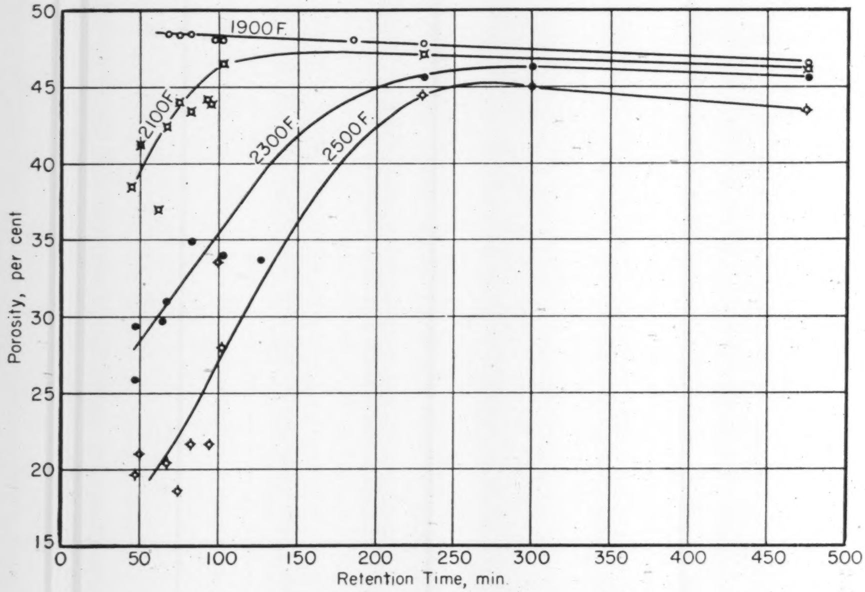

FIG. 8.—Effect of Retention Time on Porosity of Lime.
Temperatures Represent Muffle Temperatures.

Further studies of the mechanism of slaking are planned as a part of the complete program. Pending the completion of these studies, the activity index as mentioned above forms a usable index to some of the characteristics of the limes, although its purely empirical foundation must not be overlooked.

Relation between Burning Conditions, Activity, and Porosity:

Muffle temperatures were controlled at a series of fixed temperatures, namely, 1900, 2100, 2300 and 2500 F. The overall retention time in the kiln was varied from a low of about 46 min. to a high of 474 minutes.

The properties of the quicklimes produced are given in Table I with the data grouped in accordance with the control temperature of the muffle. All calcinations numbered from 19-1 to 19-8 inclusive had a muffle control temperature of 1900 F., all numbered 21-1 to 21-11 had a muffle control temperature of 2100 F., etc. In each group, the several calcinations are arranged in order of decreasing retention time.

Inspection of Table I discloses that the properties of surface area, porosity, and activity are closely correlated. High values of surface area are always associated with high values of porosity and with high activity. This correlation is better shown by Figs. 6 and 7 where activity and surface area are plotted against porosity.

The effect of burning temperature on the properties of the quicklime cannot be completely separated from the effect of retention time. Figure 8 is a plot of porosity against retention time for the limes produced at the four different muffle temperatures. Figure 9 is a similar plot of activity coefficient, ΔT_5, against retention time. From these figures it is apparent that both porosity and activity decrease with an increase in burning temperature at all retention times. This relationship has frequently been noted in the static calcination of commercial limestones.

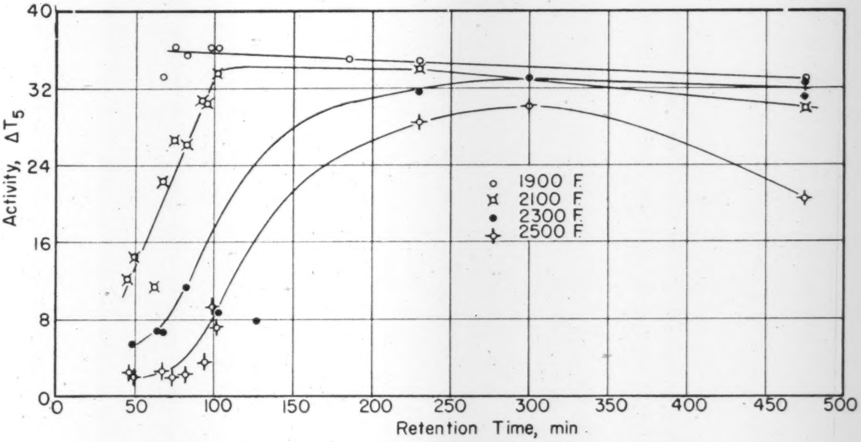

FIG. 9.—Effect of Retention Time on the Activity of Lime. Temperatures Represent Muffle Temperatures.

The normal concept of the relation of time of burning to the properties of the lime is that any increase in retention time beyond that required for complete calcination causes shrinkage of the lime and decreased porosity due possibly to the fluxing action of impurities. It is tactily assumed that the formation of this flux is a slow process and that time is essential to the attainment of maximum shrinkage of the lime.

However, since the material used in this investigation was high-purity calcite, no fluxing action was possible. Nevertheless, limes were produced which were inactive, of low porosity, and to which the commercial teminology "dead-burned" could be applied.

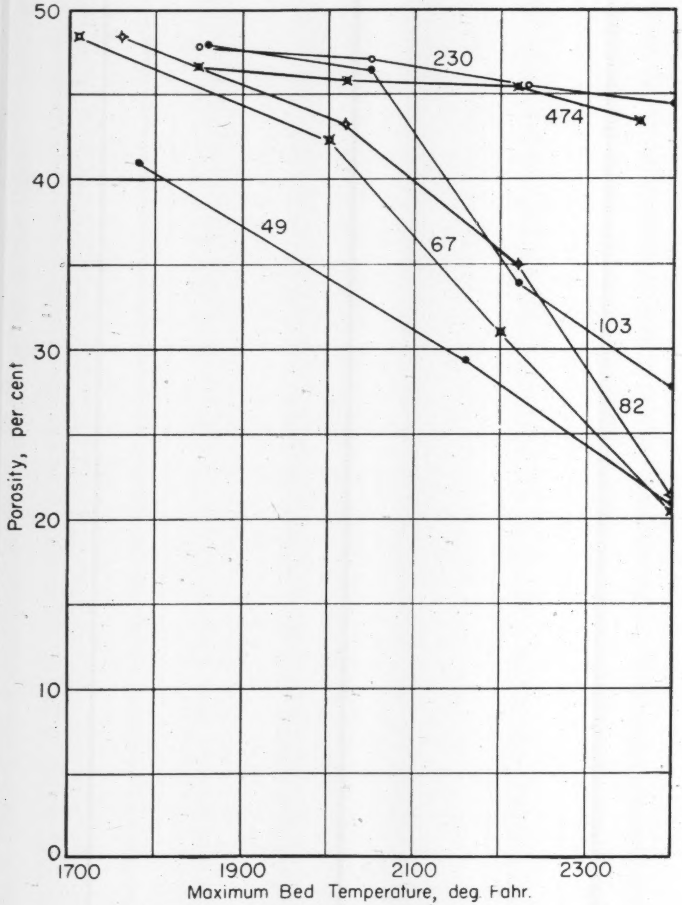

FIG. 10.—Effect of Maximum Bed Temperature on Porosity of Lime. Numbers Represent Retention Time in Minutes.

With the exception of the limes produced at the very lowest burning temperature, those produced by keeping the retention time short (50 to 100 min.) are all less porous, less active, and have less surface area than those produced with retention times of 200 min. or more (see Figs. 10 and 11). The data indicate that, for each burning temperature, an optimum retention time may be located which produces greatest porosity and activity. If the material is held in the kiln for a longer time than this optimum, the porosity decreases, but at a very slow rate. On the other hand, if the retention time is less than the optimum, the porosity and activity may be greatly decreased, particularly at the higher temperatures. Further study of the data indicates that the rate of heating of the material, either prior to calcination or during calcination, or both, is an important factor. A decomposition pressure of one atmosphere is attained with calcite at 1647 F. (897 C.) under equilibrium conditions (7). It may be assumed, as a first approximation, that under the non-equilibrium conditions in the experimental kiln the decomposition is substantially complete at 1700 F. Using the temperature distribution measurements made with the probe thermocouple, together with the calculated rate of progress of the material through the kiln, it is possible to calculate the time required for the material in the kiln to reach 1700 F. This calculation has been made for all burns. The results are given in the fourth column of Table I.

The relation between the porosity of the lime and the time required for the material to reach 1700 F. is shown in Fig. 12. This indicates that if the time to reach 1700 F. with calcite of this size is 35 min. or more, the resulting product is highly porous and active. On the other hand, if the time required to reach 1700 F. is less than 35 min., the porosity may be greatly reduced. The results appear to be almost independent of the ultimate burning temperature. Physical limitations prevent the material from reaching 1700 F. (and complete calcination) in less than 29 min. when the muffle control is operating at 1900 F. Hence all limes produced at this control temperature are highly porous and

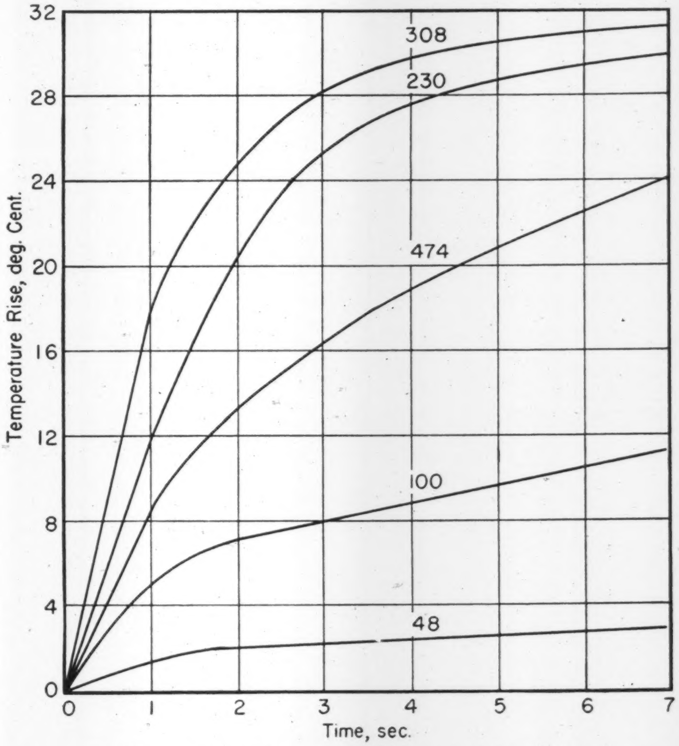

FIG. 11.—Effect of Retention Time on Activity of Lime.
Maximum Kiln Temperature, 2400 F.
Numbers Represent Retention Time in Minutes.

active. With a muffle control temperature of 2500 F., it is possible to reach a material temperature of 1700 F. in 14 min., and the limes so produced are of relatively low porosity and very inactive.

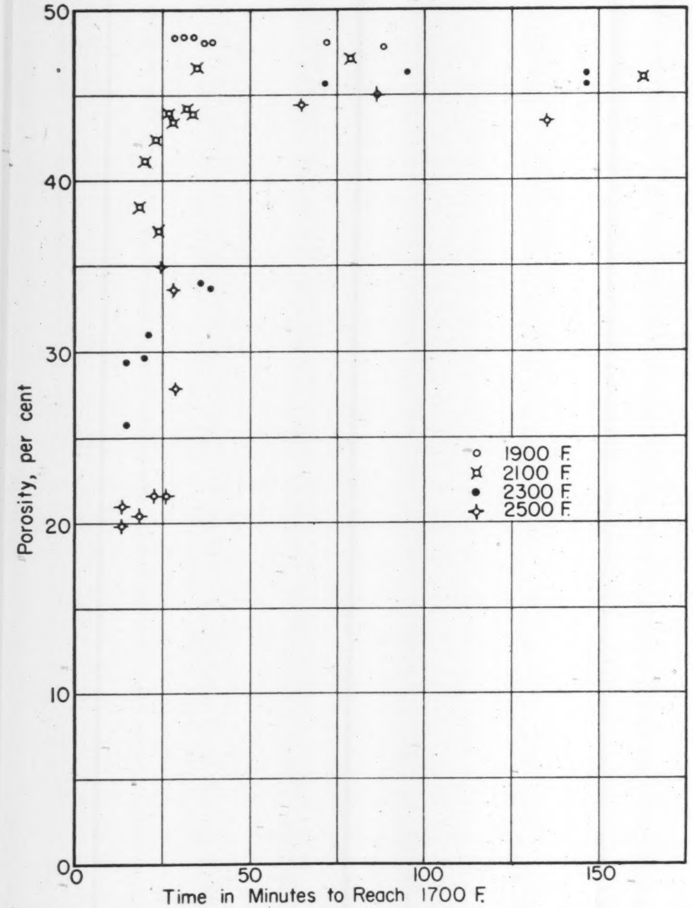

FIG. 12.—Relation between Porosity of Lime and the Time Required for the Material to Reach 1700 F.

Temperatures Represent Muffle Temperatures.

The porosity (and activity) of quicklimes apparently is a function of two factors. The first of these is the rate of heating prior to and during calcination. The second factor is concerned with the maximum temperature to which the material is exposed subsequent to calcination and the time at which it is held at this elevated temperature. It has been reported by Zimens (8) that the CaO structure "loosens" at 1200 C. (2192 F.). On the other hand, von Adrenne, Endell and Lehman (9) report that the fine structure of ignited sintered dolomite is unchanged at temperatures up to 1400 C. (2552 F.) but collapses at higher temperatures. The possibility exists, therefore, that excessive thermal vibration caused by holding material for an extended time at elevated temperatures may give rise to a loosened, more porous structure than would be formed at the same temperature but with lesser time to permit the loosening of the structure.

Further investigation is necessary to determine which of the two factors is the more important. Under the experimental conditions used in this investigation the first mentioned factor, the rate of heating prior to and during calcination, appears to be the controlling factor.

CONCLUSIONS

1. The porosity, surface area, and activity of quicklimes prepared from calcite are closely related, high values of porosity are associated with high values of surface area and with high activity.

2. With equal retention times in the kiln, higher temperatures of burning produce limes with lower porosity and activity. This effect is more pronounced at short retention times.

3. An optimum retention time which produces highest porosity and activity may be found for each burning temperature.

4. The rate of heating before and during calcination has a much greater effect on the porosity of quicklime than either the maximum temperature or the total retention time in the kiln subsequent to calcination.

Acknowledgment:

This paper is the result of work conducted under a fellowship sponsored by the National Lime Association at the Massachusetts Institute of Technology.

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DISCUSSION

Mr. Victor J. Azbe[1] (*by letter*).—To the writer, calcination was the main item of interest for just about thirty years. So, he feels qualified, in a manner, to offer some comments, although reluctantly, being that he is not in entire agreement with the authors.

As a first item, he does not believe that a rotating furnace is suitable for calcination experiments requiring exactness, whether or not it is internally or externally heated. The tendency to size segregation is too pronounced, particularly when the size range is as great as in this case, 10 to 20 mesh, the smallest only one eighth the weight of the largest.

Then, even though the authors swept the furnace with an inert gas, within the active portion of the bed CO_2 concentration was likely to be high and calcination retarded, so material passing through was, for reasons of segregation, of CO_2 concentration and varied size, unequally exposed to calcining force and subsequent overheating.

One important feature which offsets the above objections to some extent is that temperature was determined of the bed within the bed. These temperatures were very high and much higher than one is likely to encounter in rotary kiln practice.

Compared to a rotary kiln, preheating was extremely rapid, calcination period greatly prolonged and cooling very slow. Time of total retention was from $\frac{3}{4}$ to 8 hr. The ratio of hot zone time to total time was also greater. Still penetration rate zone of calcination was infinitesimal.

One needs to refer to the experiments by C. C. Furnas of the U. S. Bureau of Mines who states that with limestone, the penetration of line of calcination is constant for any given surrounding temperature. His penetration rates were:

Surrounding Temperature, deg. Fahr.	1590	1765	1930	2100	2260
Penetration, in. per hr.	0.1	0.2	0.4	0.8	1.6

It will be noted that calcination rate doubles for about every 170 F. of temperature rise.

These rates are greatly more than the authors obtained. For part of this there is an explanation and we believe that calcination rate is more a matter of surface temperature than of surrounding temperature since there may be a high surrounding temperature attendant by low radiation heat transfer and surface conductance rate.

However, the authors took the temperature of the bed. That is virtually the surface temperature of the lime, so why was such long retention time necessary for such small particle sizes. Granted that their bed may have been of higher CO_2 concentration but even then, if calcination had not started until 1650 F., it should have been complete before 1800 F. for their shortest retention time.

The only explanation that we see is that calcite was used for the stated reason of "purity" when it exactly should not have been used for reasons of "odd behavior". And even if the calcite was used, and it certainly deserves a study, conclusions should not have been formulated to apply to lime generally. Nothing much of calcite has a bearing on lime due to its calcining resistivity and long retention time necessary, during which the lime of the calcite, pure as it may be, is affected by time-temperature elements. The writer has first pointed out the peculiarity of calcite in the May 1926 issue of *Rock Products*.

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[1] President, Azbe Corp., St. Louis, Mo.

As to the authors first three conclusions given at the end of their paper, they have been known for a long time. As to the fourth, on the other hand, which is the only one that is novel, we emphatically disagree. Granted that there is a possibility that it would apply to calcite due to its resistivity to calcination and high temperatures necessary, it would not apply to any limestone of our knowledge.

The authors say, "The rate of heating before and during calcination has a much greater effect on the porosity of quicklime than either the maximum temperature or the total retention time in the kiln subsequent to calcination."

Our statement is that rate of heating before has no effect whatsoever and that calcination time or temperature have only a very minor and practically unimportant effect because almost all calcination of limestone as such is "low-temperature calcination." As long as there is calcination, temperature of the lime remains low except only for the small resistive fractions for which a limited terminal temperature build up of short duration is necessary.

It is this temperature build up, if unnecessarily long and particularly if unnecessarily high, that affects the lime in its degree of cellular consolidation, that is, shrinkage, hard burning, specific gravity gain or whatever one may call it, and attendant onto this effects on hydration and other characteristics.

That this is so is completely logical and so evident that proof can be noted through observation of any lime draw of any kiln. Further proof would hardly be necessary, but to again recheck conclusions based on tests made over the many years past, the following was done.

Three samples of limestone of varied characteristics were selected. Indiana porous "A," Missouri denser "B," and a limestone of pronounced sintering tendency "C". These were sawed into 1 in. cubes and a hole drilled for the insertion of a thermocouple. This was done because in such tests we find it better not to put too much reliance on furnace temperature but rather to base it more on the actual temperature of the specific sample and better yet, in the case of larger samples, base it on two temperatures—surface and center.

Six samples, two of each, were inserted into a Burrell furnace which was regulated for a slow heating rate. At 1900 F. of sample temperature one set was removed. At 2550 F. the others were removed. Results are given below:

Sample Stone	Gravity of Stone	Gravity of Lime	Consolidation, per cent	Hydration		Weight Loss
				Time	Temperature, deg. Fahr.	
SLOW HEATING RATE—1900 F. SAMPLES						
No. 1—A—Indiana....	2.248	1.284	1.23	8.5	25.5	43.5
No. 2—B—Missouri...	2.443	1.499	7.64	4.5	27.6	43.3
No. 3—C—Sintering..	2.254	1.42	7.91	4.0	22.8	42.0
Retention Time 1500 to 1900 F. = 68 min.						
SLOW HEATING RATE—2550 F. SAMPLES						
No. 4—A—Indiana....	2.260	1.489	14.48	49.5	15.4	43.7
No. 5—B—Missouri...	2.445	1.81	17.59	22.0	23.1	43.8
No. 6—C—Sintering..	2.278	2.16	39.19	23.0	2.7	42.3
Retention Time 1500 to 2550 F. = 98 min.						

Specific gravity of the stone and lime was determined through forcible immersion in mercury through the use of the arrangement shown in the accompanying Fig. 13.

Under hydration, the temperature rise is given and the time in minutes at which it was attained. Figure 14 presents com-

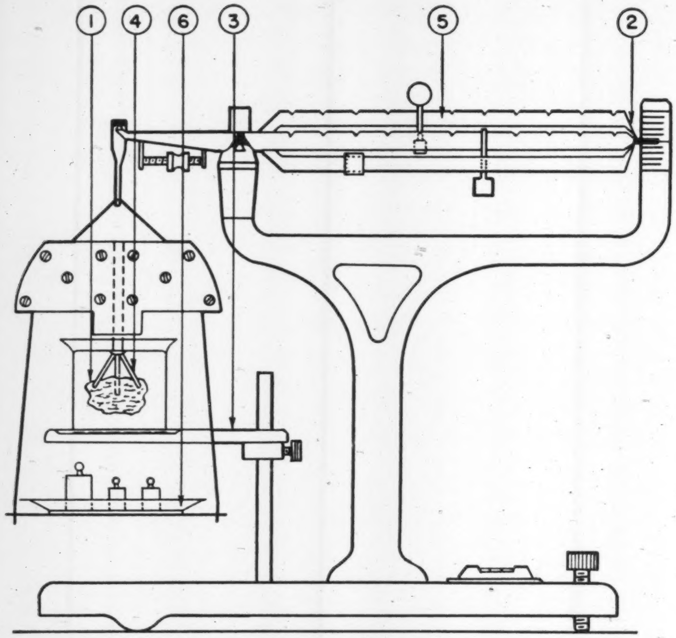

FIG. 13.—Lime Gravity Apparatus.

General Procedure

1. Determine weight of sample 1 in air. This is weight "A."
2. Set and lock pointer 2 at zero balance position and adjust platform 3 so mercury level is at gage mark on immersion prong 4.
3. Release 2 and balance by weighting system 5. Leave weights in this position for rest of determination.
4. Place Sample 1 under prong and immerse in mercury.
5. Repeat step 2 above.
6. Release 2 and balance by weights at 6. This is weight "B."
7. Specific gravity of sample $= \dfrac{\text{Specific gravity of Mercury} + \text{“A”}}{\text{“A”} + \text{“B”}}$

   Specific gravity of mercury at 68F. (20C.) = 13.5462

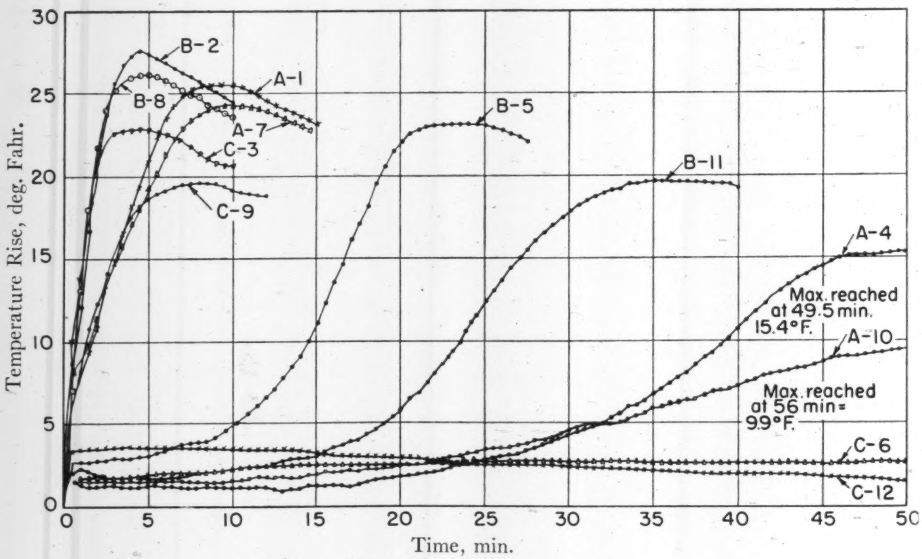

FIG. 14.—Hydration Characteristics of Lime.

plete time-temperature relation during hydration.

In the above tabulation it will be noted that there was but a slight difference in the degree of calcination between 1900 and 2550 F. samples and that consolidation affecting hydration rate took place in the main above 1900 F. Lime of 1900 F. was very active while that of 2550 F. was quite sluggish. Maximum furnace temperature for 1900 was 2200 F. and for 2550 F. samples it was 2660 F.

The test was repeated in a similar manner but at a higher heating rate with the following results:

Sample Stone	Gravity of Stone	Gravity of Lime	Consolidation, per cent	Hydration		Weight Loss
				Time	Temperature, deg. Fahr.	
FAST HEATING RATE—1900 F. SAMPLES						
No. 7—A—Indiana....	2.242	1.328	4.65	9.5	24.3	43.6
No. 8—B—Missouri...	2.444	1.558	9.60	4.5	26.2	42.5
No. 9—C—Sintering..	2.256	1.60	18.82	8.0	19.6	42.4
Retention Time 1500 to 1900 F. = 48 min.						
FAST HEATING RATE—2550 F. SAMPLES						
No. 10—A—Indiana...	2.241	1.618	22.13	56	9.9	43.8
No. 11—B—Missouri..	2.444	2.0	31.40	35	19.7	43.9
No. 12—C—Sintering.	2.262	2.373	45.1	5	3.6	42.6
Retention Time 1500 to 2550 F. = 78 min.						

In this case maximum furnace temperature for the 1900 F. sample was 2330 F., and through the entire period it was higher than during the slow heating rate test. It is plain to see the consolidation that took place in either of the tests was in the outer portions of the sample towards the surface subsequent to calcination.

The low temperature necessary, the rapid penetration zone of calcination at the relatively very low average furnace temperature prevailing during calcination period of these tests are given below, with penetration rates for the same temperature many times higher than those the authors of the paper obtained.

TIME-TEMPERATURE AND PENETRATION RATE 1500 TO 1900 F. PERIOD OF SAMPLE TEMPERATURE.

	Slow Heating	Fast Heating
Elapsed time 1500 to 1900 F., min...	68	48
Furnace temperature at 1900 F. center of sample temperature, deg. Fahr....	2200	2330
Average furnace temperature over above time, deg. Fahr....	1647	1775
Cube size, in....	0.916	0.916
Total penetration, in....	0.458	0.458
Penetration per hour, in....	0.413	0.57

The authors at one point criticize the normal concept of relation of time of burning to the properties of the lime. They point out that their high purity calcite where no fluxing action was possible also overburned. In answer to this one may say that there is no prevailing misconception and that everyone in the lime industry knows that any lime can be hard burned. But, there is a big difference between over burning of lime such as "B" of these tests, which is one of the purest in this country, and either "A" or "C". The Missouri sample may be hard burned, highly consolidated, of greatly affected hydration rate, but after it is hydrated it will still be lime of high available CaO content. This will be considerably less so in the case of "A," while the sintering "C," while not exceptionally impure, if hard burned becomes useless to any chemical process, and in all these cases it is the overheating subsequent to calcination that affected the lime.

The writer presented a paper before this Society in 1939 as part of the Lime Symposium.[2] In all main respects conclusions therein still hold good today.

MESSRS. J. A. MURRAY, H. C. FISCHER, AND D. W. SABEAN (*authors' closure*).—The differences in behavior between calcite and limestone were recognized by the authors. The rate of calcination differs from that of Furnas, both because of a difference in raw material and because of the difference in experimental techniques. In regard to the application of the results to commercial stones, we personally considered this to be questionable until similar studies on more normal stones had been completed. However, we have been informed by one lime producer that he has already made a practical application of the principles outlined in our paper, and has successfully produced a quality of lime which he had hitherto been unable to produce.

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[2] V. J. Azbe, "Fundamental Mechanics of Calcination and Hydration of Lime and Methods of Control," Symposium on Lime, p. 88, Columbus Regional Meeting, Am. Soc. Testing Mats. (1939). (Symposium issued on separate publication *STP No. 40.*)

EXPLORATORY TESTS TO DEVELOP A METHOD FOR DETERMINING THE AIR CONTENT OF HARDENED CONCRETE*

By Alexander Klein[1], David Pirtz[1], and Milos Polivka[1]

Synopsis

This paper describes two methods for determining the air content of hardened concrete: (1) an absolute-volume method, which consists in pulverizing the concrete to a given fineness and then, making allowance for evaporable water and impermeable voids, computing the air content from the difference between the over-all volume of the concrete specimen and its absolute volume after pulverizing; and (2) a vacuum-saturation method in which the air content is computed from the change in weight of a specimen due to saturation. The saturation of a standard-cured specimen is accomplished by first subjecting the specimen to vacuum and then saturating it with water under pressure.

Air contents as obtained by the two methods are compared to the air content of the corresponding fresh concrete as determined by the A.S.T.M. Tentative Method of Test for Air Content (Volumetric) of Freshly Mixed Concrete (C 173 – 42 T)[2]. The absolute-volume method gives lower values of air content than the vacuum-saturation method. Both methods appear to be of equal reproducibility. The vacuum-saturation method is simpler and more rapid. Results show that the air content of concrete decreases with age and that the type of agent used has an effect on this reduction in air content.

In view of the current interest in methods of determining the air content of hardened concrete,[3] the authors believe it propitious at this time to present the results of an investigation of this problem, even though the tests were of an exploratory nature and somewhat limited in scope.

This study lead to the development of two methods: (1) an absolute-volume method, and (2) a vacuum-saturation method.

In considering the resistance of concrete to alternate cycles of freezing and thawing, the effective air is that which is present in the hardened concrete rather than that which is present in the fresh mix. Based on determination of air in fresh concrete, it has been found in previous investigations that the resulting hardened concretes vary in resistance to freezing and thawing, depending on the air-entraining agent employed.[4] The results of this investigation indicate that the air content of hardened concrete decreases with age and that the rate of this decrease varies with the type of agent employed. This change in volume of air content is probably due to the filling of the capillary channels during the hydration of the cement paste.

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[*] Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Research Engineer, Lecturer in Civil Engineering, and Instructor in Civil Engineering, respectively, University of California, Berkeley, Calif.

[2] 1949 Book of A.S.T.M. Standards, Part 3, p. 810.

[3] Robert P. Vellines and Thomas Ason, "A Method for Determining the Air Content of Fresh and Hardened Concrete," *Journal*, Am. Concrete Inst., Vol. 20, May, 1949; *Proceedings*, Vol. 45, pp. 665–671; G. J. Verbeck, "The Camera Lucida Method for Measuring Air Voids in Hardened Concrete," *Journal*, Am. Concrete Inst., Vol. 18, May, 1947; *Proceedings*, Vol. 43, pp. 1025–1039.

[4] Raymond E. Davis, "Entrained Air Beneficial in Freezing and Thawing Tests," Symposium on Concretes Containing Air-Entraining Agents, *Journal*, Am. Concrete Inst., Vol. 15, June, 1944; *Proceedings*, Vol. 40, p. 552.

MATERIALS AND SAMPLE PREPARATION

Local natural sand and gravel, 0 to $\frac{3}{4}$ in., and type II portland cement were used in all mixes. Two air-entraining agents, designated A and B, were employed. The concretes were of two cement contents, 3 and $5\frac{1}{4}$ sacks per cubic yard, and were of like consistency as measured by the Powers remolding apparatus.

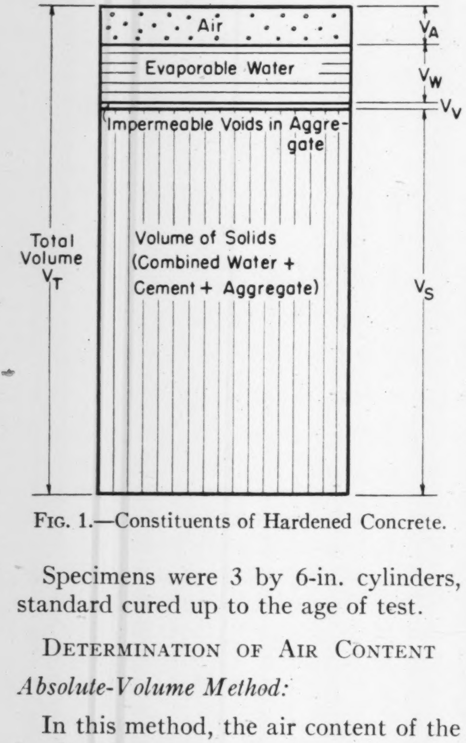
Air
Evaporable Water
Impermeable Voids in Aggregate
Volume of Solids (Combined Water + Cement + Aggregate)
Total Volume V_T
V_A
V_W
V_V
V_S

FIG. 1.—Constituents of Hardened Concrete.

Specimens were 3 by 6-in. cylinders, standard cured up to the age of test.

DETERMINATION OF AIR CONTENT

Absolute-Volume Method:

In this method, the air content of the hardened concrete was computed from the following formula:

$$\text{Per cent of air} = \frac{V_T - V_S - V_W - V_V}{V_T} \times 100$$

where:

V_T = over-all volume of specimen,
V_S = absolute volume of solids in the specimen (combined water plus cement plus aggregate),
V_W = volume of evaporable water, and
V_V = volume of impermeable voids in the aggregate.

These various components of hardened concrete are shown in Fig. 1.

The over-all volume V_T was computed from the difference in weight, as measured in air and in water, after the specimen had been removed from standard curing.

The absolute solid volume V_S was computed from the specific gravity of the solids in the specimen and its weight after oven-drying at 230 F. for 7 days. For the specific gravity determination, the specimens were crushed and pulverized to a specific surface of 2900 sq. cm. per g.[5]

The volume of evaporable water V_W in the concrete was obtained from the difference in weight of the specimen in its standard-cured condition and in its oven-dry condition.

The volume of impermeable voids in the aggregate, V_V, was computed from the difference between the apparent specific gravity and the specific gravity of the solids in the aggregate. The "apparent specific gravity" of a piece of aggregate is the ratio of its oven-dry weight, in grams, to its volume, including its impermeable voids, in milliliters; while the "specific gravity" is the ratio of the oven-dry weight, in grams, of a piece of aggregate to its solid volumes, in milliliters, after it has been pulverized to eliminate all impermeable voids. For the aggregate used in this investigation, it was found that the volume V_V was so small as to render unnecessary its inclusion in the computation.

Vacuum-Saturation Method:

By this method, the air content of the hardened concrete was computed from:

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[5] This determination was made by the air-permeability method, employing a porosity of 0.505.

$$\text{Per cent of air} = \frac{V_G}{V_T} \times 100$$

where:

V_T = over-all volume of specimen, and

V_G = volume of water gained by saturating a standard-cured specimen.

Volume of water gained, V_G, was the difference in weight of a standard-cured specimen before and after saturation. The process of saturation was as follows: The specimen was first oven-dried and then placed in a pressure vessel in which a vacuum of 29 in. of mercury was maintained for 2 days; thereafter while maintaining vacuum, water was slowly introduced into the vessel until it was completely filled; vacuum was then discontinued, and a pressure of 50 psi. was applied for 2 days, followed by a pressure of 200 psi. for an additional 6 hours. This procedure was employed in the belief that it would produce the maximum degree of saturation.

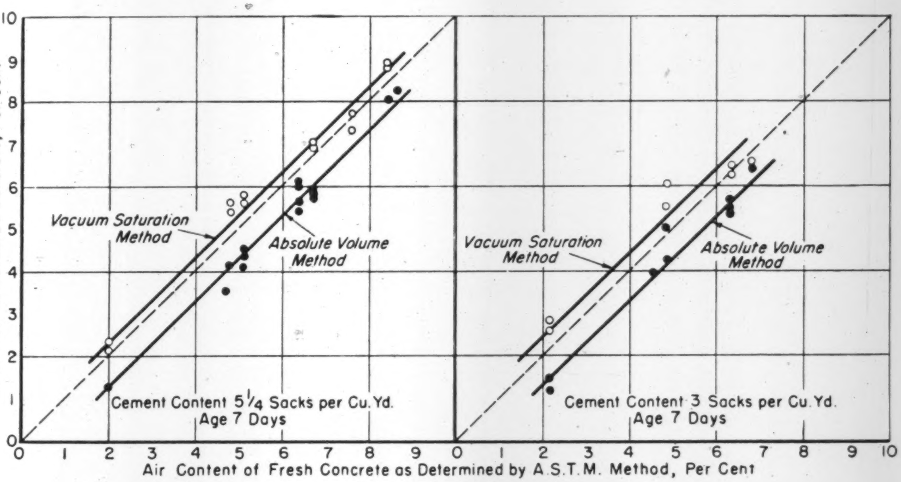

FIG. 2.—Comparison of Air Content of Fresh and Hardened Concrete.

DISCUSSION OF RESULTS

In Fig. 2 is shown a comparison of the air content of hardened concrete at age 7 days, as determined by the two methods, with that of fresh concrete. The air content of the hardened concrete, as determined by the absolute-volume method, is lower than that of the fresh concrete, while, as determined by the vacuum-saturation method, it is higher than that of the fresh concrete. The lower values obtained by the absolute-volume method may be due to the reduction of air voids through gel growth in the cement paste. Since vacuum will desiccate the capillaries in the concrete, it is to be expected that a volume of water larger than that of the air voids will be necessary to saturate the specimen; the resulting air content of the hardened concrete as determined by the vacuum-saturation method will therefore appear to be larger than that of the corresponding fresh concrete. For both the 3- and the 5¼-sacks-per-cubic-yard mixes, the difference between the air contents of the hardened concretes as determined by the two methods is approximately 1 per cent.

The tests indicate that the air content of hardened concrete decreases with age. Figure 3 shows a typical comparison at the ages of 7, 14, and 28 days, for a 5¼-sacks-per-cubic-yard mix, with nominal air contents of 5, 6½, and 8½ per cent. It is believed that this change with age is due to gel growth, with corresponding reduction in the size of capillaries, in the hardening cement paste. In general, the air content of hardened concrete containing agent A decreases at a more rapid rate than that containing agent B, and this decrease is more rapid between 0 and 14 days than between 14 and 28 days. At 28 days the compressive strength of concrete containing agent A was 32 per cent higher than that of concrete containing agent B. From previous tests it was found that, for the same air content, concretes containing agent A were much more resistant to freezing and thawing than those containing agent B, although the results shown in Fig. 3 indicate a larger decrease in air content at 28 days for concretes containing agent A than for those containing agent B. It would appear, therefore, that the resistance of concrete to freezing and thawing depends not only on the air content but also on the volume of the capillaries.

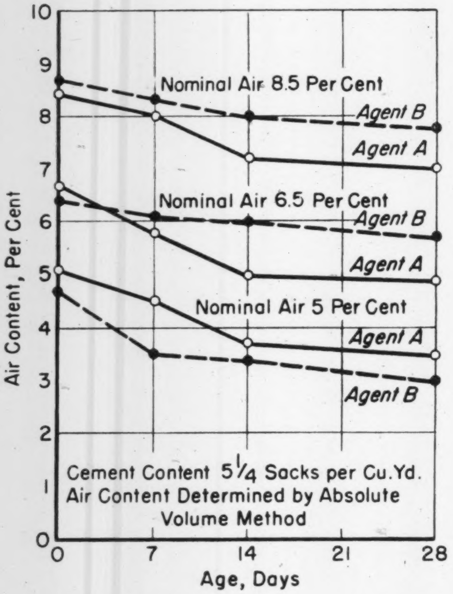

FIG. 3.—Effect of Age and Agent on the Air Content of Hardened Concrete.

In general there is good correlation between the air content of hardened and fresh concretes, regardless of richness of mix or type of agent. In making comparisons, the probable error in the air-content determinations should be taken into consideration. In determining the air content of hardened concrete, the probable error, in percentage of air, is about 0.3, which is of approximately the same precision as is obtained with methods used to determine the air content of fresh concrete. Thus, in a comparison of the air content of fresh and hardened concrete, the combined probable error, in percentage of air, will be approximately 0.5.

CONCLUSIONS

In determining the air content of hardened concrete, it appears that one of the two methods herein described is as reproducible as the other and that the reproducibility of either appears to equal that of methods for determining the air content of fresh concrete.

The vacuum-saturation method is simpler and more rapid, although the air contents obtained by this method appear to be slightly high. In the absolute-volume method, there are more opportunities for errors than in the vacuum-saturation method. As determined by the absolute-volume method, air contents of hardened concretes correlate better with those of corresponding fresh concretes, and, as would be expected, are lower in the hardened concrete than in the fresh concrete.

The air content of hardened concrete decreases with age, probably due to gel growth in the cement paste, this decrease being more rapid at early than at later ages.

Acknowledgments:

The authors are indebted to Raymond E. Davis of the University of California for his counsel in planning the test program. Acknowledgment is made to John Gilcrest and Ernest Clay, senior students in Civil Engineering at the University of California, for performance of the tests.

DISCUSSION

MR. BRYANT MATHER.[1]—We have not endeavored to approach the question of the air content of hardened concrete by either of these methods.

We have, however, done a considerable amount of work with the microscope approach. Our difficulties in making comparisons between air content of hardened concrete and air content of plastic concrete appear to have arisen from the nonsimilarity of the actual concrete samples used in the two tests.

The amount of consolidating effort used, the influence of specimen molds in causing edge effects, the differences in segregation of materials, and loss of water by bleeding in containers of different depths in which the concrete remains for different lengths of time, seemed to be responsible for a variable difference between determinations of air content of plastic and hardened concrete that we are, as yet, unable to eliminate entirely. We are, therefore, not yet able to tell, when our results on hardened concrete differ from those on presumably similar plastic concrete, how much they indicate a change in air content of the concrete and how much they indicate original differences between the samples tested.

We are inclined to think that the concrete we look at in the hardened condition has had its air content changed from a presumably similar sample tested in the plastic condition, principally by factors other than merely age and development of gel, although they too have some influence. My only point is to indicate some of the difficulties that may be brought into this matter from factors other than normal changes in the cement paste.

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[1] Chief, Special Investigations Branch, Concrete Research Div., Waterways Experiment Station, Jackson, Miss.

METHOD OF PETROGRAPHIC EXAMINATION OF AGGREGATES FOR CONCRETE*,[1]

By Katharine Mather[2] and Bryant Mather[2]

Synopsis

A detailed description of the method for petrographic examination of aggregates for concrete as it has been developed for use by the Concrete Laboratories of the Corps of Engineers is presented. The differences in procedure which depend upon the nature of the sample submitted and the purposes of the examination are described. Suggestions are made concerning the features to be delineated, the amount of material to be examined, and the organization and presentation of the results of the examination. It is hoped that the information presented will serve to clarify the concept of what is meant by petrographic examination of aggregates and, perhaps, ultimately, contribute to greater uniformity in the making of such examinations.

There does not appear to have been a previous presentation of a detailed outline of the method by which a qualified petrographer may apply the knowledge and techniques of geology to the examination of the various types and conditions of material that are considered for use as aggregates for concrete. There are an increasing number of engineering organizations that are undertaking the application of petrographic methods to the study of concrete aggregate, an increasing number of petrographers who are being confronted with the need to examine concrete aggregates, and an increasing number of engineers who are called upon to evaluate petrographic data. It is therefore felt desirable to present a detailed statement of the method for petrographic examination of aggregates for concrete as it has been developed for use by the seven concrete laboratories of the Corps of Engineers.

In a previous paper[3] the use of petrographic methods for the identification of reactive constituents in concrete aggregate was discussed. The merits of petrographic examination of concrete aggregate materials and the results of such examinations have been dealt with in a number of papers, many of which are included in the bibliography.

The method as set forth here represents the most recent product of work begun more than a decade ago by Elliot P. Rexford, the first concrete laboratory petrographer of the Corps of Engineers, whose last paper[4] gives a

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Approved for presentation and publication by the Office, Chief of Engineers, U. S. Army.

[2] Head, Petrography Section and Chief, Special Investigations Branch, respectively, Concrete Research Division, Waterways Experiment Station, Jackson, Miss.

[3] B. Mather, "Petrographic Identification of Reactive Constituents in Concrete Aggregates," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, pp. 1120–1127 (1948).

[4] E. P. Rexford, "Some Factors in the Selection and Testing of Concrete Aggregates for Large Structures," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 187, No. 3, pp. 395–402 (1950). (Technical Publication 2837 H).

valuable summation of his long experience. To him and to the other petrographers with whom the authors have been associated, appreciation is expressed for much assistance; they include: W. L. Donn, Rosalind Ravitch, George Townsend, Louis Moyd, Pauline Moyd, Jeanne Weiser, O. G. Alessio, N. B. Dodge, R. A. Dunkerley, and R. V. Tye.

This method has been editorially modified from that published as CRD-C 127–50 in the Handbook for Concrete and Cement issued by the Waterways Experiment Station.

EXAMINATION AND CLASSIFICATION

This method outlines the procedures to be employed in the petrographic examination of samples representative of materials proposed for use as aggregates in concrete. The scope of such an examination may be limited as in the case of preliminary examinations, or complete, as in the case of final examinations.

Preliminary Examination:

The preliminary examination is designed to assist the field geologist in judging which of apparently suitable aggregate sources contain material of sufficiently promising quality to warrant further and more complete investigation. Such preliminary examinations should be used as a part of the process of elimination in the survey of economically available aggregate sources when other evidence is not available as a basis for sound initial judgment of quality. The value of a preliminary petrographic examination will be dependent largely upon the adequacy of the samples provided, upon the data on the deposit (amount of material, predictable difficulties in working, etc.) which are provided with the samples, and on the proposed use of the material. The results of such an examination should not be used alone as a basis for final acceptance of a material.

Complete Examination:

The complete petrographic examination is designed to constitute an integral part of the comprehensive study of the suitability of an aggregate material for the use for which it is proposed. The value of such an examination will be greatly influenced by the adequacy of the procedures used in obtaining the samples which are submitted for examination.

The specific procedures employed in the petrographic examination of any sample of aggregate material depend to a large extent both on the purpose of the examination and on the nature of the sample submitted.

Purposes:

The purposes to be served by petrographic examination are as follows:

(*a*) *Properties.*—The delineation of all physical and chemical properties that may be observed by petrographic means which bear on the quality of the materials for their intended use.

(*b*) *Classification.*—The description and classification of materials present in samples of aggregate will logically include identification of the rocks in the sample. Identification is usually a necessary step towards recognition of the properties which may be expected to influence the behavior of the materials in their intended use. It is not an end in itself. Rock of any type may perform well or poorly as concrete aggregate depending on the physical conditions of the aggregate, and on its physical and chemical properties as compared to the matrix in which it may be placed.

The data developed by a petrographic examination can provide a basis for predicting quality, provide an explanation of the results obtained from performance tests, and give information on factors not at present capable of being investigated by performance tests. The usefulness of any petrographic examination to the agency requesting it will depend largely on three factors:

1. The representativeness of the samples provided.

2. The information accompanying the samples, which should include the location, type of sampling, extent of deposit (if known), name of geologic formation (if known).

3. The petrographer's degree of understanding of the information needed by the agency requesting the examination. It should be the responsibility of the agency requesting the examination to make its needs clearly and specifically known to the laboratory. It should be the responsibility of the petrographer to understand and make use of the information available to him. This information will frequently include the results of elementary tests on the sample, chemical tests of potential reactivity, and freezing and thawing of concrete containing the aggregates under test.

APPARATUS AND SUPPLIES

Specimen Preparation:

The apparatus and supplies consist of:

1. Diamond saw, preferably with 14-in. blade,
2. horizontal grinding wheel, preferably 16 in. in diameter,
3. polishing wheel, preferably 8 to 12 in. in diameter,
4. abrasives: silicon carbide grit No. 100, No. 240, FFF; emery such as American Optical Co. M-303, and M-304,
5. prospector's pick,
6. microscope slides, clear, noncorrosive, 25 by 45 mm. in size,
7. Canada balsam, neutral, in xylol,
8. xylol,
9. glycol phthalate,
10. small laboratory oven,
11. plate-glass squares about 12 in. on an edge for thin section grinding,
12. thin-section holder,
13. Jones riffle sampler with pans,
14. micro cover glasses, noncorrosive, square 12–18 mm., 25 mm., etc., and
15. Plattner mortar.

Specimen Examination:

The apparatus and supplies consist of:

1. Polarizing microscope with mechanical stage; low-, medium-, high-power objectives, objective centering devices; eyepieces of various powers; full- and quarter-wave compensators; quartz wedge,
2. microscope lamps,
3. stereoscopic microscope with paired objectives and paired oculars to give final magnifications from about 6 × to about 60 ×,
4. magnet, preferably alnico, or an electromagnet,
5. needleholder and points,
6. dropping bottle, 60-ml. capacity,
7. Petri culture dishes,
8. smooth straight-pointed forceps,
9. lens paper,
10. immersion media, n = 1.410 to n = 1.785 in steps of 0.005,
11. Abbé refractometer,
12 sodium arc lamp,
13. zirconium arc lamp,
14. Wratten light filters,
15. counter,
16. photomicrographic camera and accessories.

Examples of the above-listed items are shown in Figs. 1 to 8.

NOTE.—The apparatus and supplies listed above and illustrated in Figs. 1 to 8 comprise a recommended selection. It is not intended that the specific items listed and illustrated must necessarily be provided. It is however regarded as essential that an adequate selection be provided to enable the use of all of the procedures described in this method for the examination of the particular types of samples that are to be studied.

Samples:

Samples for petrographic examination should be taken by or under the direct supervision of a geologist familiar with the requirements of aggregates for concrete and in general following the requirements of A.S.T.M. Methods of Sampling Stone Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 – 48).[5] The exact location from which the sample was taken, the geology of the site, and other pertinent data should be submitted with the sample.

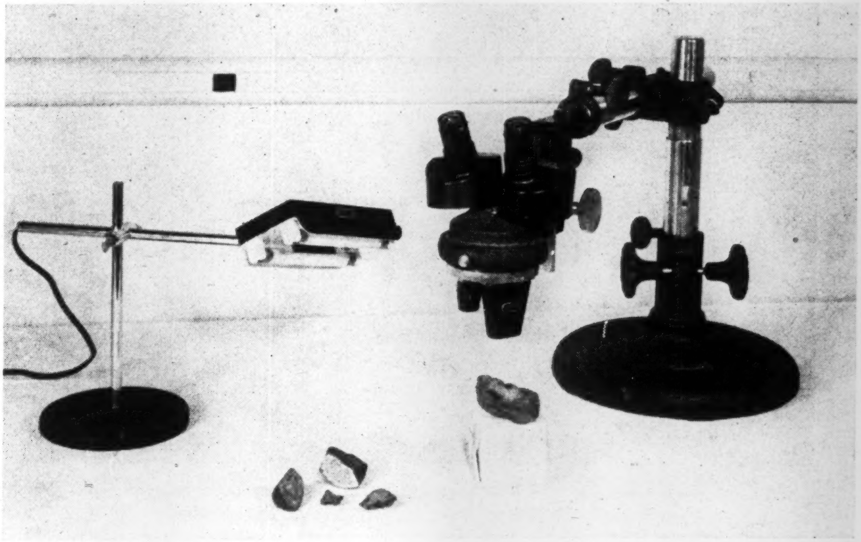

FIG. 1.—Equipment for Examination of Samples at Low Magnifications.

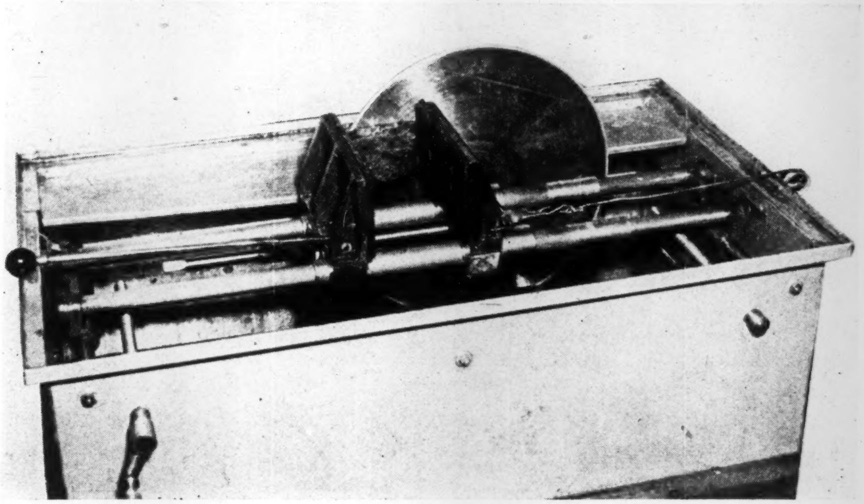

FIG. 2.—Diamond Saw.

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[5] 1949 Book of A. S. T. M. Standards, Part 3, p. 758.

Undeveloped quarries should be sampled by means of cores, drilled through the entire depth expected to be productive of sound material. Drilling of such cores should be in a direction which is essentially normal to the dominant structural feature of the rock. Massive material may be sampled by "NX" cores. Thinly bedded or complex material should be represented by cores

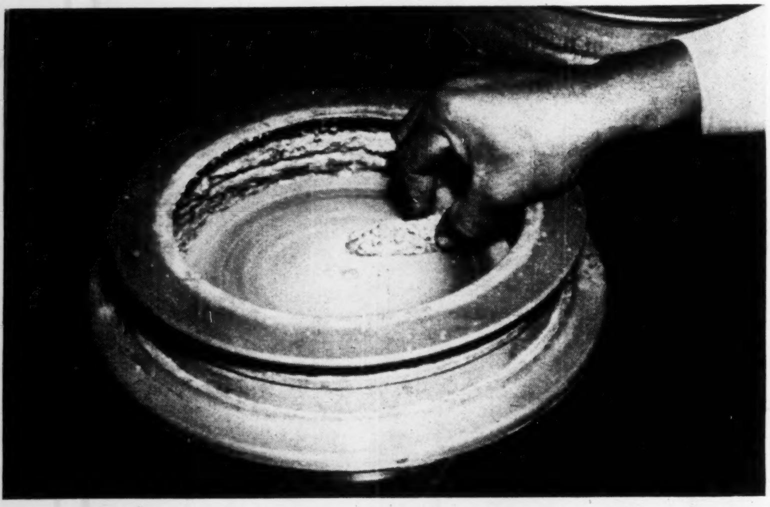

FIG. 3.—Horizontal Lap for Grinding and Smoothing Sawed Surfaces of Rock.

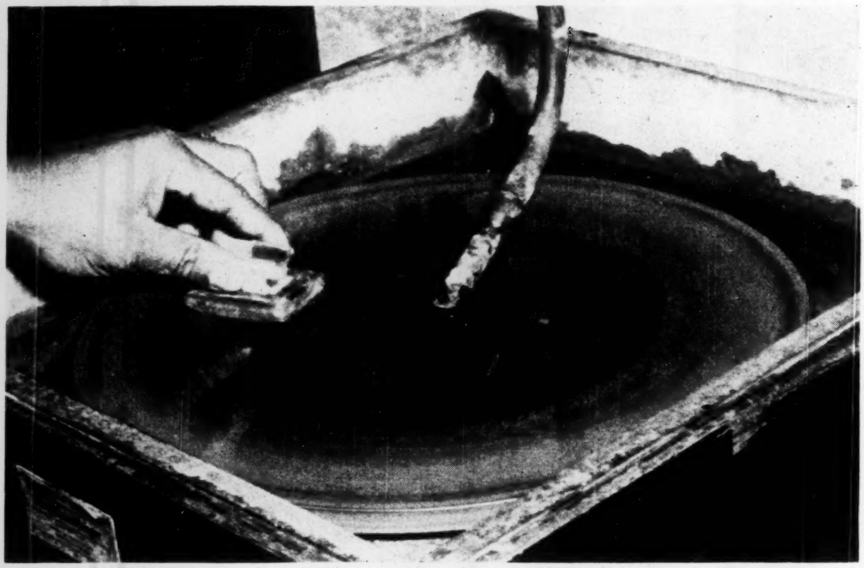

FIG. 4.—Grinding Thin Section Blank on Horizontal Lap.

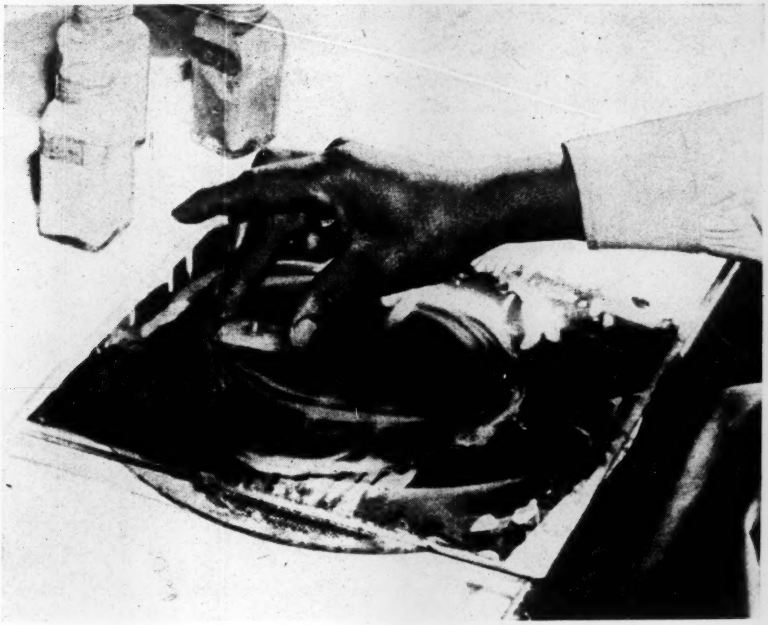

FIG. 5.—Final Grinding of Thin Section on Glass Plate.

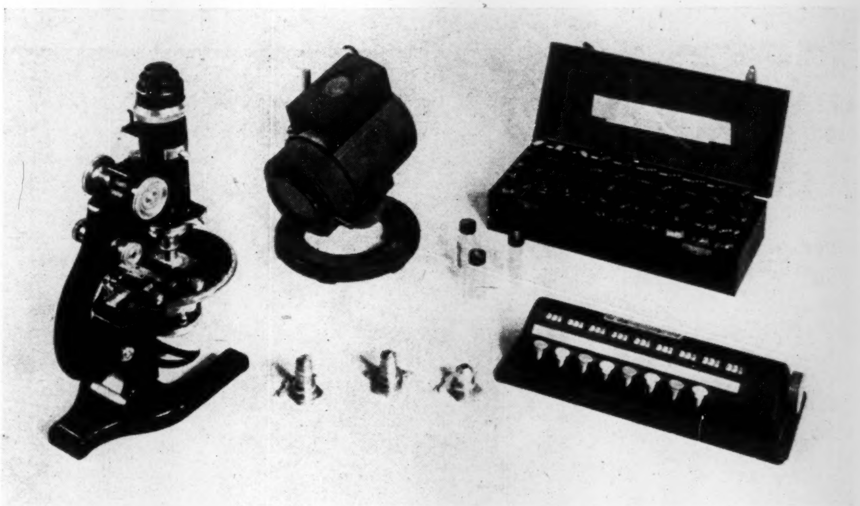

FIG. 6.—Petrographic Microscope for Examination of Thin Sections or Powder Mounts, Index Media, Counter.

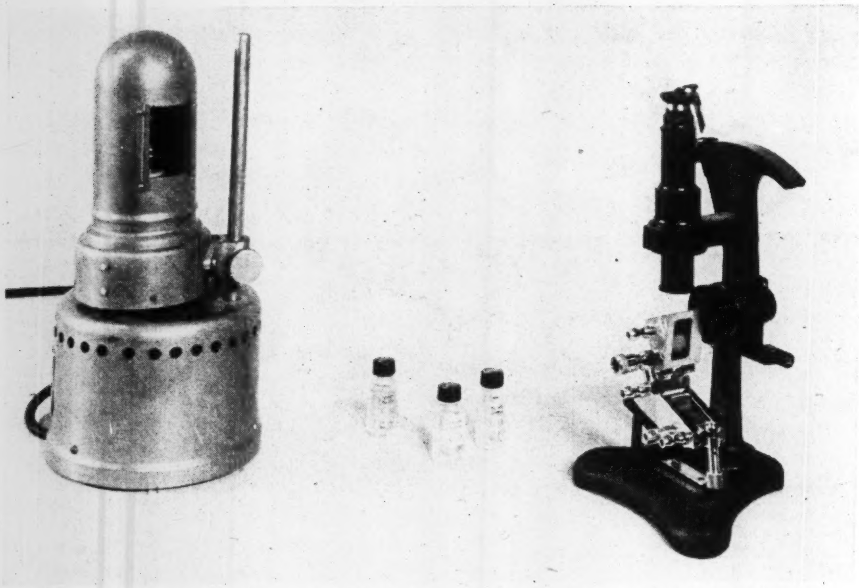

FIG. 7.—Sodium Arc Lamp and Abbé Refractometer for Calibrating Index Media.

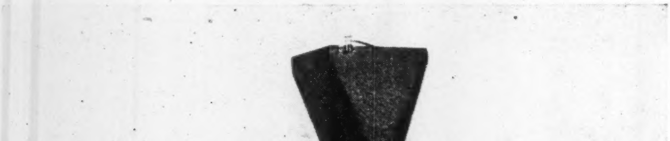

FIG. 8.—Photomicrographic Camera in Use Over Petrographic Microscope.

not less than 4 in. in diameter. There should be an adequate number of cores to cover the limits of the deposit proposed for the work under consideration. The entire footage of the recovered core should be included in the sample and accurate data given as to elevations, depths, and core losses.

Operating quarries and operating sand and gravel deposits in which stock piles of the material produced are available, should be represented by not less than 100 lb. or 300 pieces, whichever is larger, of each size of material to be examined. Samples from stock piles should be composed of representative portions of larger samples collected with due consideration given to segregation in the piles.

Exposed faces of non-producing quarries, where stock piles of processed material are not available, should be represented by not less than 4 lb. from each distinctive stratum or bed with no piece weighing less than 1 lb.; or by drilled core as described in the first paragraph of this section.

Undeveloped sand and gravel deposits should be sampled by means of test pits dug to the anticipated depth of future economic production. Samples should consist of not less than the following quantities of material which are representative of the deposit:

Sieve Size	Quantity	
	Lb.	Pieces
6 to 12 in.	...	[a]
3 to 6 in.	...	300[a]
1½ to 3 in.	400	
¾ to 1½ in.	200	
No. 4 to ¾ in.	100	
Sand	50	

[a] Not less than one piece from each apparent type of rock.

The amount of material actually studied in the petrographic examination will be determined by the nature of the examination to be made and the nature of the material to be examined, as discussed in the next section.

TYPES OF MATERIAL

As previously noted, the specific procedures used in any petrographic examination depend to a large extent both on the purpose of the examination and on the nature of the sample submitted. The procedures to be selected will be those which will give adequate information on the properties and classification of the materials contained in the sample examined. When the intended use of the aggregate is known, the specific methods used in a petrographic examination will depend on the type of sample and the complexity of the sample. The samples considered in this test method may be classified into two groups.

Natural Gravel and Natural Sand:

This group includes those materials in which the particle shape and particle surface of the finished graded aggregate result chiefly from natural processes.

Drilled Core, Ledge Rock, Crushed Stone, and Manufactured Sand:

This group includes those materials in which the particle shape and particle surface of the finished graded aggregate result chiefly from the manufacturing process.

Methods for the examination of samples of the first and second groups will be discussed in more detail. Many of the procedures described for the examination of materials of one group will be applicable to some samples in the other group.

PROCEDURE FOR PETROGRAPHIC EXAMINATION OF NATURAL GRAVEL AND SAND

Samples:

Samples of gravel and natural sand for petrographic examination should be sieved according to A.S.T.M. Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 - 46)[6] to provide samples of each sieve size. In the case of sands an additional portion should then be sieved, without being washed and tested according to A.S.T.M. Method of Test for Amount of Material Finer Than No. 200 Sieve in Aggregates (C 117 - 49)[7] in order to provide a sample of the material passing the No. 200 sieve. The results of the sieve analysis of each sample made according to A.S.T.M. Method of Test for Sieve Analysis of Fine and Coarse Aggregate (C 136 - 46)[6] should be provided to the petrographer making the examination and used in calculating results of the petrographic examination. Each sieve fraction should be examined separately, starting with the largest size available. Rocks are more easily recognized in larger pieces; the breakdown of a heterogeneous type present in the larger sizes may have provided particles of several apparently different types in the smaller sizes; some important and easily confused types may be recognizable using the stereoscopic microscope if they are first recognized and separated in the larger sizes, but may require examination using the petrographic microscope if they are first encountered in the smaller sizes. The number of particles of each sieve fraction examined will be fixed by the required precision of determination of the less abundant constituents.[8] It is believed that at least 300 particles of each sieve fraction should be identified and counted in order to obtain reliable results. Precise determinations of small quantities of an important constituent will require counts of larger samples.

Examination of Natural Gravel:

Coatings.—The pebbles should be examined to establish whether exterior coatings are present. If they are, it should be determined whether the coatings consist of materials likely to be deleterious in concrete (opal, gypsum, easily soluble salts, organic matter). It should also be determined how firmly the coatings are bonded to the pebbles.

Rock Types.—The sieve fraction should be sorted into rock types by visual examination. If all or most of the groups present are types easily identifiable in hand specimen by examination of a natural or broken surface, and by scratch and acid tests, no further identification may be needed. Fine-grained rocks which cannot be identified megascopically or which may consist of or contain constituents known to be deleterious in concrete should be checked by examination with the stereoscopic microscope. If they cannot be identified by that means, they should be examined using the petrographic microscope. The amount of work done in identifying fine-grained rocks should be adapted to the information needed about the particular sample. Careful examination of one size of a sample, or information from previous examination of samples from the same source, will usually make apparent the amount of additional detailed microscopic work required to obtain information adequate for the purpose.

Condition.—The separated groups belonging to each rock type should be examined to determine whether a further separation by physical condition is necessary. If all of the particles of a rock type are in a comparable condition, that fact is noted. More frequently particles in several degrees of weathering will be found in a group. They should be sorted into categories based on condition and on the expectation of comparable behavior in concrete. The types of categories intended are: (*a*) Fresh, dense; (*b*) moderately weathered; (*c*) very weathered: or (*a*) Dense; (*b*) porous (or porous and friable). It is usually impractical to recognize more than three conditions per rock type, and one or two may be sufficient. An important constituent present in large quantities may sometimes require separation into four groups by condition. The conspicuous example is chert when it is the major constituent of a gravel sample. It may be present as dense, unfractured chert; as vuggy chert; as porous chert; and as dense but highly fractured chert. The determination of which of these four conditions characterizes a particle may be expected to have an important influence on prediction of the behavior of the particle in concrete.

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[6] 1949 Book of A.S.T.M. Standards, Part 3, p. 763.

[7] *Ibid*, p. 748.

[8] Assuming that the field sampling and laboratory sampling procedures are accurate and reliable, the number of particles examined, identified, and counted in each sieve fraction will depend on the required accuracy of the estimate of constituents present in small quantities. The numbers given in this method are minimal. They are based on experience and on statistical considerations discussed in the following two references:

G. G. Simpson and A. Rowe, "Quantitative Zoology," McGraw-Hill Co., Inc., New York, N. Y., pp. 182–185 (1939).

A. L. Dryden, Jr., "Accuracy in Percentage Representation of Heavy Mineral Frequencies" *Proceedings*, Nat. Academy Sciences U. S., Vol. 17, No. 5, May, 1931, pp. 233–238.

Sequence of Examination of Sizes.—Examination of the largest size usually indicates the major groups of constituents in a gravel and the principal considerations which are likely to affect the suitability of the aggregate for the intended use. The other sieve fractions should then be examined in order of diminishing size, separated into rock types, and counted. As new groups appear, they should be identified and counted. Changes in the condition of each group from sieve fraction to sieve fraction should be recorded. Ordinarily it will not be necessary to examine in detail the material passing the No. 4 sieve in a gravel sample, unless it amounts to more than 5 per cent of the nominal No. 4 to $\frac{3}{4}$-in. size range, or unless inspection suggests that it contains constituents which may be expected to have an important effect on the suitability of the aggregate for the intended use.

Record.—Notes should be taken during the examination. Each rock-type should be described; the relevant features may include:

1. Particle shape,
2. particle surface,
3. grain size,
4. texture and structure, including observations of pore space, packing of grains, cementation of grains,
5. color,
6. mineral composition,
7. significant heterogeneities,
8. general physical condition of the rock-type in the sample, and
9. presence of constituents known to cause deleterious chemical reaction in concrete.

Particle counts should be recorded so that tables can be made for inclusion in the report. When the examination has been completed, the notes should contain enough information to permit the preparation of tables and descriptions. Tables should be prepared showing the composition and condition of the samples by sieve fractions, and the weighted average composition, based on the gradation of the sample as received and on the distribution of constituents by sieve fractions. Descriptions of constituent groups should be prepared containing the relevant features among those listed in the preceding paragraph.

Examination of Natural Sand:

The procedure for the examination of natural sand is similar to that for the examination of gravel, with the modifications necessitated by the differences in particle size.

Sizes Coarser than No. 30.—Each sieve fraction present that is coarser than the No. 30 sieve should be reduced in the Jones riffle until a split or splits containing at least 300 particles are obtained. The reduced sample of each sieve fraction should be examined, and its constituents identified, and counted using the stereoscopic microscope. It is convenient to spread out the sample in a flat-bottomed glass dish such as a Petri dish and manipulate the grains with a forceps and dissecting needle. The identification of grains in the coarser sand sizes is often easier when the grains are just submerged in water. The submergence lessens reflection from the outer surfaces and may show diagnostic features which cannot be seen when the grains are dry. There are exceptions to this generalization. Where identification is difficult, the examination includes examination of the natural surface (dry and wet), examination of a broken surface (dry and wet), and scratch and acid tests. Only after all of these steps have been taken and the grain is still unidentified should the petrographer resort to the petrographic microscope. Grains which cannot be identified using the stereoscopic microscope or which are suspected of consisting of or containing substances known to react deleteriously in concrete should be set aside to be examined with the petrographic microscope. If the question of reaction with the minor alkalies of portland cement is important in the examination of the sample, certain additions to the procedure are indicated. If the coarser sand sizes contain fine-grained possibly glassy igneous rocks, several typical particles of each variety of such rocks should be selected for more thorough examination. The petrographer should determine the presence or absence of glass by crushing typical grains and examining them in immersion media using the petrographic microscope. In difficult or especially important cases, it may be necessary to break suspected grains, make immersion mounts of part of the grain and a thin section of another part. Where the sand contains chert and the potential reactivity of the chert is an important consideration, a number of chert particles from the fractions retained on the No. 30 sieve should be set aside for determinations of the index of refraction.

Sizes Finer than No. 30.—The sieve fractions finer than the No. 30 sieve should each be reduced in a Jones riffle to about 4 or 5 g. The volume will usually be less than a level teaspoonful. In some gradations the fractions retained on the No. 100 and No. 200 sieves may be present in such small amounts that reduction is unnecessary. These splits should be further reduced on a miniature Jones riffle or by coning and quartering with a spatula on a clean sheet of paper. A representative portion of each reduced split should be mounted in immersion oil on a clean glass slide and covered with a clean cover glass. No entirely satisfactory method of reducing a split to about 300 grains is known. The reduced split can be sampled by spreading it in a thin layer on glass or clean paper, dragging the end of a dissecting needle moistened in immersion oil through the sample, and transferring the grains that adhere to the needle to a drop of immersion oil on a clean slide. If this is done carefully, a fairly representative sample will be obtained. If the dissecting needle is magnetized steel, a concentration of magnetic minerals may result. It is usually necessary to make several mounts of the No. 50 and No. 100 sieve fractions to obtain at least 300 grains of each. The index of the immersion oil should be selected to make the identification of the important constituents as easy and as definite as possible. Practice at the Waterways Experiment Station is to use an immersion oil with an index of or just below the lower index of quartz (1.544). The slide should be mounted on a petrographic microscope equipped with a mechanical stage. Several traverses should be made, and each grain which passes under the cross hair should be identified and counted. Care should be taken to move the slide on the north-south adjustment between traverses so that no grain should be counted twice. Each sieve fraction passing the No. 30 and retained on the No. 200 sieve should be examined in this way. Ordinarily, the material passing the No. 200 sieve is mounted on a slide following the procedure described above, examined using the petrographic microscope, and its composition estimated. If an unusually large amount of this size is present, or if it contains constituents which may be expected to have an important effect on the suitability of the aggregate for the intended use, it should be counted.

PROCEDURE FOR PETROGRAPHIC EXAMINATION OF DRILLED CORE, LEDGE ROCK, CRUSHED STONE, AND MANUFACTURED SAND

Drilled Core:

Each core should be examined and a log prepared showing footage of core recovered, core loss and location; location and spacing of fractures and parting planes; lithologic type or types; alternation of types; physical condition and variations in condition; toughness, hardness, coherence; obvious porosity; grain size, texture, variations in grain size and texture; type or types of breakage; presence of constituents capable of deleterious reaction in concrete. If the size of the core permits, the probability that the rock will make aggregate of the required maximum size should be considered. If the surface of the core being examined is wetted it is usually easier to recognize significant features and changes in lithology. Most of the information usually required can be obtained by careful visual examination, scratch and acid tests, and hitting the core with a hammer. In the case of fine-grained rocks, it may be necessary to examine parts of the core using the stereoscopic microscope, or to prepare thin sections of selected portions. Some considerations and procedures are more applicable to particular rock-types than to others. Ordinarily, the layered rocks considered for concrete aggregate will be limestone, and occasionally metamorphic rocks, such as phyllite or schist. One of the most important questions arising in the examination of limestone is that of the presence, type, and distribution of argillaceous impurities. Limestones which contain intercalated thin beds of soft shale may make suitable sources of aggregate if the shale is so distributed that it does not prevent manufacture of the required maximum size, and if the shale can be eliminated or reduced in processing. Where argillaceous impurities are present, it should be determined whether they actually consist of clay minerals or of other minerals in clay sizes. If they do consist of clay minerals, it should be established whether the clay minerals include members of the montmorillonite group (swelling clays).[9] In the examination of fine-grained igneous rocks, particular attention should be directed to the nature of the groundmass. This examination should include determination of the presence or absence of opal, chalcedony, natural glass and clay minerals of the montmorillonite group; if any of these are found the amount should be estimated; if natural glass is found the type should be determined.

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[9] G. F. Loughlin, "Usefulness of Petrology in the Selection of Limestone," *Rock Products*, Vol. 31, No. 6, March 17, 1928, pp. 50–59.

Ledge Rock:

The procedure in examination should be the same as for core samples to the extent that the spacing of samples and size of the individual pieces allow. If the sample consists of a relatively large quantity of broken stone produced by blasting, it is desirable to inspect the whole sample, estimate the relative abundance of rock types or varieties present, and sample each type before further processing. Subsequent procedure should be the same as given below for crushed stone.

Crushed Stone:

The procedure for examination of crushed stone should be similar to that for core, except that necessary quantitative data should be obtained by particle counts of the separated sieve fractions

TABLE I.—CALCULATION OF RESULTS OF PARTICLE COUNTS.

Constituents	Composition of Fractions Retained on Sieves Shown Below							
	¾-in.		½-in.		⅜-in.		No. 4	
	Number of Particles	Per Cent	Number of Particles	Per Cent	Number of Particles	Per Cent	Number of Particles	Per Cent
A_1	250	50.0	200	40.0	150	30.0	50	10.0
A_2	50	10.0	100	20.0	125	25.0	100	20.0
A_3	10	2.0	50	10.0	75	15.0	100	20.0
B_1	107	21.4	70	14.0	62	12.4	32	6.4
B_2	76	15.2	53	10.6	19	3.8	87	17.4
B_3	...	...	20	4.0	43	8.6	96	19.2
C_1	5	1.0	5	1.0	20	4.0	20	4.0
C_2	2	0.4	2	0.4	6	1.2	10	2.0
C_3	...	...	...	...	...	...	5	1.0
Totals	500	100	500	100	500	100	500	100
Individual per cent retained on sieve		17.4		32.6		29.5		20.5

	Weighted Percentages of Constituents in Each Sieve Fraction				Weighted Composition of Sample	
	¾-in.	½-in.	⅜-in.	No. 4		
A_1	8.7	13.0	8.9	2.1	32.7	64.6 (Total A)
A_2	1.7	6.5	7.4	4.1	19.7	
A_3	0.4	3.3	4.4	4.1	12.2	
B_1	3.7	4.6	3.7	1.3	13.3	31.8 (Total B)
B_2	2.6	3.5	1.1	3.6	10.8	
B_3	...	1.3	2.5	3.9	7.7	
C_1	0.2	0.3	1.2	0.8	2.5	3.6 (Total C)
C_2	0.1	0.1	0.3	0.4	0.9	
C_3	...	...	...	0.2	0.2	
Total in sieve fraction	17.4	32.6	29.5	20.5		
Total in sample, condition 1					48.5	
Total in sample, condition 2					31.4	
Total in sample, condition 3					20.1	

Manufactured Sand:

If no samples of the rock from which the sand was produced are available, the examination procedure should be similar to that for natural sand, with particular emphasis on the amount and extent of fracturing developed by the milling operations.

CALCULATIONS

The composition of each sieve fraction of a heterogeneous sample and the weighted average composition of the whole sample should be calculated as follows:

1. The composition of each sieve fraction shall be expressed by summing the total number of particles of that fraction counted, and calculating each constituent in each condition as a percentage of that total (amount, as number of particles in per cent, in each sieve fraction). It is convenient to calculate and record the percentages to tenths at this stage. An example of these calculations is given in the upper half of Table I.

2. The percentage by weight of the sieve fraction in the whole sample (individual percentages retained on consecutive sieves) shall be obtained from the gradation of the sample as determined by A.S.T.M. Method (C 136-46).[6] (Individual per cent retained on sieve in Table I.)

3. By multiplying the percentage of the constituent in the sieve fraction, determined under (*1*), by the percentage of the sieve fraction in the whole sample, obtained in (*2*), the percentage in the whole sample of that constituent in that size shall be obtained (weighted percentage of constituents in sieve fraction, Table I). It is convenient to calculate and record these percentages to tenths.

4. By adding the weighted percentages of each constituent in each sieve fraction, the weighted percentage of each constituent in the whole sample shall be obtained (see under weighted composition of sample in Table I).

5. A table shall be constructed to show the composition of each sieve fraction and the weighted composition of the whole sample. Values shall be reported to the nearest whole number. Constituents amounting to 0.5 per cent or less of a sieve fraction or of the whole sample shall be reported as traces. Table II is an example constructed from the data obtained in Table I. As a convention, the total in each sieve fraction and the total in the whole sample shall each be 100 per cent without the traces. Difficulties in abiding by this convention can usually be avoided by grouping minor constituents of little engineering importance. It is preferable to tabulate constituents known to react deleteriously in concrete so that their distribution will be apparent from inspection of the table, even though the amount in the whole sample or in any fraction is very small.

TABLE II.—COMPOSITION AND CONDITION OF AN AGGREGATE SAMPLE (TABLE CONSTRUCTED FROM CALCULATIONS SHOWN IN TABLE I).

Constituents	Amount, as number of particles in per cent							
	In Fractions Retained on Sieves Shown Below[a]				In Whole Sample[b]			
	¾ in.	½ in.	⅜ in.	No. 4	Condition 1	Condition 2	Condition 3	Totals
A.........	62	70	70	50	33	20	12	65
B.........	37	29	25	43	13	11	8	32
C.........	1	1	5	7	2	1	tr	3
Total...	100	100	100	100	..	..	..	100
Weighted average, condition 1...					48	...	...	...
Weighted average, condition 2....					....	32	...	...
Weighted average, condition 3....					....	...	20	...

[a] Based on count of 500 particles in each sieve fraction.

[b] Based on gradation of the sample as received, and on the distribution of constituents by sieve fractions shown at the left above. (If the petrographic report forms part of a complete investigation of the sample, including a report of the gradation, the gradation need not be shown. If the petrographic report is to be submitted alone, the gradation of the sample should be included with it.)

REPORT

The report of the petrographic examination should consist of two principal parts, the summary and the detailed report as indicated in Appendix A. The summary report customarily consists of two paragraphs which summarize, respectively, the essential data needed to identify the sample as to source and proposed use, and the features of the material as revealed by the examination. The complete report records the test procedures employed, the data developed on the composition of the material, and a description of the nature and features of each important constituent of the sample, accompanied by such tables and photographs as may be required to present adequately the findings of the examination.

Since it is improbable that the engineer who must make the ultimate decision as to the suitability of a material for use as concrete aggregate will be a geologist or petrographer, it is highly desirable that the terminology used in the summary report be simplified to the greatest extent compatible with an adequate presentation of the data. A list of rock and minerals names is given in Appendix B. It is suggested that insofar as practicable the terms used in summary reports be those included in this list.

The petrographic report should be a statement of the findings of the examination. When the sample has been found to possess properties or constituents which are known to have specific unfavorable effects in concrete those properties or constituents should be described qualitatively and, to the extent practicable, quantitatively. The unfavorable effects that may be expected to ensue in concrete should be mentioned also. When appropriate, it should be stated that a given sample was not found to contain any undesirable features. The report should not, however, contain a general statement of opinion by the petrographer concerning the quality of the material. The petrographer should avoid such general statements of opinion since they are subjective and therefore not properly a part of a report of objective findings, and since they can properly be made only when data on different materials can be compared in connection with the requirements for a specific use.

APPENDIX A

PETROGRAPHIC REPORT—SUMMARY

SYMBOL: PORT-3 6088	PROJECT: McNary Dam	DATE REPORT SUBMITTED: April 15, 1948	INITIALS: KM
SERIAL NO.: PORT-3 G-1, S-1...	SOURCE: G-1, Berrian Island, Washington Shore. S-1, 50 per cent Berrian Island, 50 per cent Jones-Scott Pit, Oregon Shore		

Sample:

On February 17, 1948, two samples, one consisting of 3 bags of natural gravel, and the other of 3 bags of sand, were received. Information accompanying the samples stated that the source of the gravel was Berrian Island, Washington Shore, and the sources of the sand were 50 per cent Berrian Island, Washington Shore, and 50 per cent Jones-Scott Pit, Oregon Shore. CRD Serial No. PORT-3 G-1 was assigned to the gravel and CRD Serial No. PORT-3 S-1 to the sand.

Summary:

Petrographic examination has been made of samples of natural gravel of 1-in. maximum size from Berrian Island, Washington Shore (PORT-3 G-1) and of sand produced by combining material from Berrian Island, Washington Shore with that from the Jones-Scott Pit, Oregon Shore (PORT-3 S-1). The coarse aggregate is tough dark rounded gravel consisting principally of basalt, with andesite, rhyolite, quartzite, quartz, chert, granite, and gabbro. The fine aggregate is composed of similar rocks and of the minerals derived from them: basalt, feldspar, quartz, ferromagnesian minerals, ore and accessory minerals, and clay. About 5 per cent of the coarse aggregate consists of rhyolite and andesite containing acid and intermediate natural glass and rhyolite containing tridymite; about 20 per cent of the coarse aggregate consists of basalt containing intermediate natural glass, with a minor amount of basalt containing basic natural glass. Some of the particles of glassy basalt also contain small amounts of secondary chalcedony and opal. Approximately 27 per cent of the fine aggregate consists of basalt, andesite, and rhyolite containing intermediate or acid glass, particles of natural glass, and chalcedony. Acid and intermediate natural glass, tridymite, chalcedony, and opal have all been shown to be reactive with the minor alkalies of portland cement.

PETROGRAPHIC REPORT—DETAILED PETROGRAPHY

SYMBOL: PORT-3 6088	PROJECT: McNary Dam	DATE REPORT SUBMITTED: April 15, 1948	INITIALS: KM
SERIAL NO: PORT-3 G-1, S-1...	SOURCE: G-1, Berrian Island, Washington Shore. S-1, 50 per cent Berrian Island, 50 per cent Jones-Scott Pit, Oregon Shore		

Test Procedure:

Representative portions of each sieve fraction of the gravel were examined and sorted megascopically and with the stereoscopic microscope. Forty-five thin sections were prepared and studied using the petrographic microscope. A large number of powders were examined using the petrographic microscope, to determine the mineral composition of particles and to determine the index of refraction of natural glass detected in the thin sections. Several photographs were taken. Representative portions of each sieve fraction of the sand were examined and sorted with the help of the stereoscopic and petrographic microscopes.

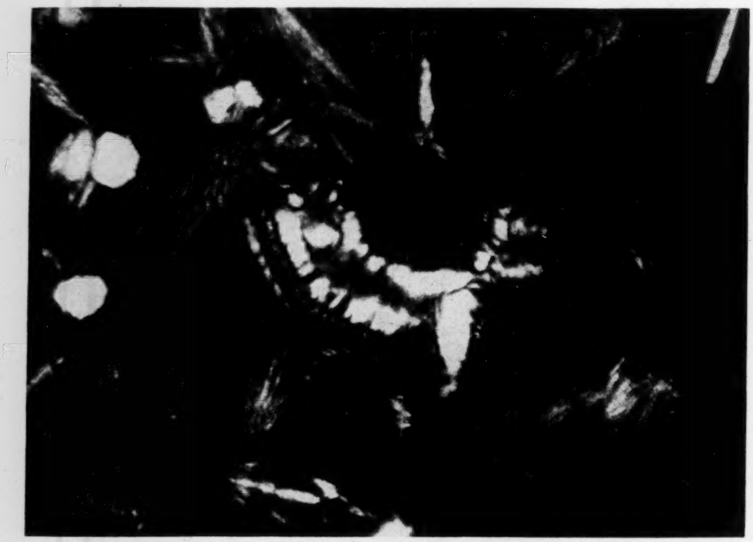


FIG. 9.—Basalt Pebble from Berrian Island, Washington Shore (Port-3 G-1).

Photomicrograph of glassy vesicular basalt (× 450, crossed nicols), showing vesicle filled with chalcedony at center. Much of the dark gray groundmass is somewhat devitrified natural glass.

Composition:

The coarse aggregate is dark rounded natural gravel, composed principally of fine-grained volcanic rocks with some quartzite, chert, quartz, mylonite, granite, and gabbro. The sand is composed of fragments of similar rocks and of the minerals derived from them. The most abundant rock-type in the gravel and the sand is basalt. About half of the basalt pebbles in the gravel contains natural glass; a few contain chalcedony or opal. Rhyolite and andesite containing glass and rhyolite containing tridymite amount to approximately 5 per cent of the sample. Almost all of the gravel is dense, tough, and unweathered. The sand is more weathered than the gravel, and many of the basalt particles are partially coated with clay.

TABLE III.—COMPOSITION OF GRAVEL PROPOSED FOR USE IN McNARY DAM (PORT-3 G-1).

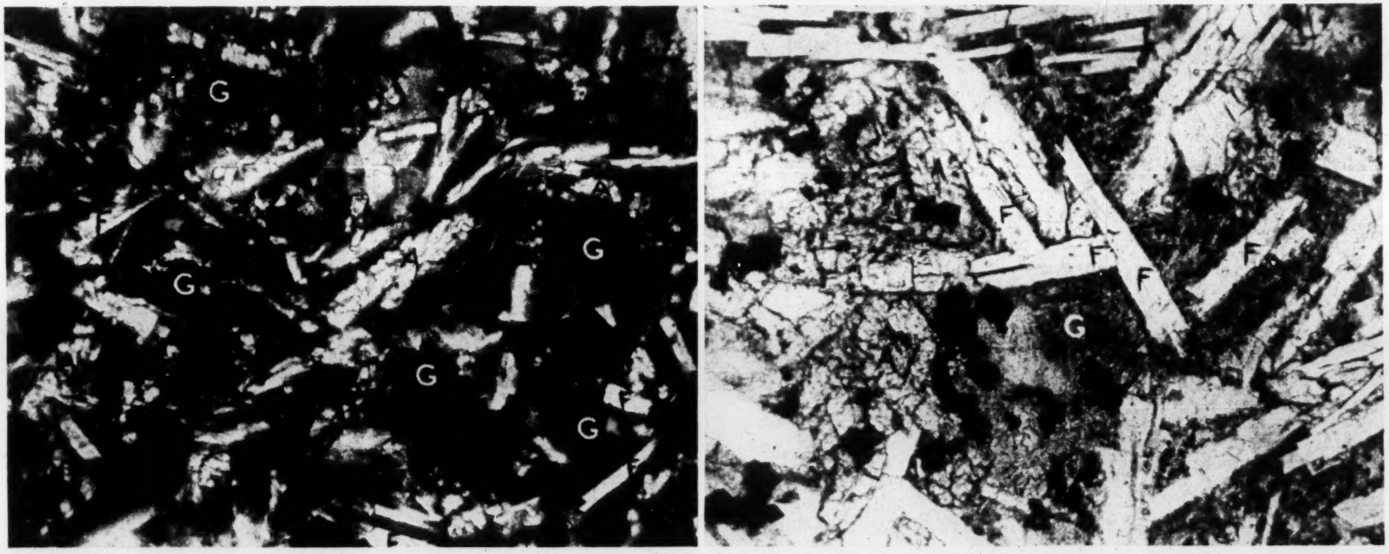
Constituents	Amount, as number of particles in per cent
Volcanic rocks without glass:	
(Basalt, andesite, rhyolite and rhyolite porphyry)	50
Volcanic rocks with glass or tridymite:	
Basalt containing glass	20
Andesite and rhyolite with glass, rhyolite with tridymite	5
Miscellaneous:	
(Granite, gabbro, quartzite, mylonite, quartz, chert)	25
Total	100

TABLE IV.—COMPOSITION OF SAND PROPOSED FOR USE IN McNARY DAM (PORT-3 S-1).

Constituents	Amount as number of particles in per cent[a]						
	In Fractions Retained on Sieves Shown Below:						In Whole Sand[b]
	No. 8	No. 16	No. 30	No. 50	No. 100	No. 200	
Volcanic rocks with glass; glass; chalcedony	39	37	32	23	8	3	27
Volcanic rocks without glass	50	44	47	11	11	10	32
Feldspar	4	5	8	47	47	56	23
Quartz	6	7	6	11	6	4	7
Ferromagnesian minerals (Pyroxene, amphibole, olivine, mica)	...	1	2	1	15	15	4
Miscellaneous (Sediments; clay; ore and accessory minerals)	1	6	5	7	13	12	7
Total	100	100	100	100	100	100	100

[a] Based on the examination of 500 particles in each sieve fraction.

[b] Based on grading of the sand as received, and on the distribution of constituents by sieve fractions shown at the left above.



(*a*) Photomicrograph of glassy basalt (× 200, plane polarized light), constituents indicated by letters: A = Augite, F = Plagioclase Feldspar, G = glass. The black polygons are ore minerals. Almost all of the medium to dark gray groundmass is intermediate natural glass.

(*b*) Photomicrograph of glassy basalt (× 200, plane polarized light). The constituents are the same as those shown at the left, but the crystals are larger and less glass is present.

FIG. 10.—Basalt Pebbles From Berrian Island, Washington Shore (Port-3 G-1)

The composition of the gravel and the sand is shown in Tables III and IV, and the constituents are described below.

Volcanic Rocks Without Glass:

A wide variety of types are included here; the pebbles range from acid types such as rhyolite, rhyolite and quartz porphyry, through andesite to basic types, principally basalt.

Rhyolite and rhyolite porphyry.—The particles in this group are rounded, usually pale in color, smooth, and hard. Most are buff, light gray, or white on freshly broken surfaces. Almost all have porphyritic texture with phenocrysts of quartz, orthoclase, or acid plagioclase in a fine-grained groundmass. In many, the groundmass consists largely of spherulites of radially crystallized feldspar and quartz; in others, the groundmass consists of a granophyric intergrowth of feldspar and quartz. All of the particles in this group are completely crystalline, and almost all are dense and tough.

Andesite and altered andesite.—Most of the pebbles in this group are rounded, greenish-gray, fine-grained, and dense. Some of them are porphyritic, with phenocrysts of intermediate plagioclase or of augite in a groundmass composed of microlites of plagioclase. The groundmass in some is felted; in others, the feldspar microlites show parallel orientation and flow structure. Several of the pebbles in this group are altered andesites, in which the original ferromagnesian minerals have been replaced by minerals of the chlorite group. All the particles in this class are completely crystalline, and almost all are dense and tough.

Basalt.—Approximately 20 per cent of the gravel consists of rounded pebbles of tough dark green, gray, or black basalt. The majority of the crystalline basalts studied in thin section are composed of laths of calcic plagioclase with interstitial pyroxene and ore minerals. A minority contain olivine as well as pyroxene. Some contain large quantities of interstitial ore minerals which make the groundmass entirely opaque and raise the specific gravity of the particles.

Volcanic Rocks With Glass or Tridymite:

This group includes pebbles of light-colored rhyolite, greenish andesite, and dark, sometimes vesicular, basalt. All of them contain materials which are known to be reactive with the minor alkalies of portland cement.

Rhyolite and andesite.—The rhyolite and and andesite are generally similar to those described in the paragraph above, but contain interstitial natural glass of intermediate or acid composition. In one thin section of a rhyolite, sparse phenocrysts of orthoclase and sodic plagioclase were scattered in a spherulitic groundmass containing small crystals of tridymite.

Basalt.—The basalt particles which contain natural glass are gray, dark green, or black. Some contain phenocrysts which can be seen with the naked eye; others are apparently structureless and break with conchoidal fracture. Some are vesicular, with the rims of the vesicles bleached; in a few the vesicles have been filled with hisingerite, chalcedony (Fig. 9), opal, or clay minerals of the montmorillonite group. The crystalline constituents of these pebbles are the same as those of the basalt without glass, but the crystals are generally smaller, and the groundmass is made up of glass (Fig. 10(*a*)) or contains patches of glass (Fig. 10(*b*)). The glass varies in color from pale yellow through tan and brown to black. It is believed that the dark color is due to minute diffused particles of opaque ore minerals. Almost all of the natural glass in the basalt pebbles has indices of refraction below 1.57; in a few particles glass of index above 1.57 was detected.

Miscellaneous:

A wide variety of rock and mineral fragments amount to approximately 25 per cent of the gravel and 7 per cent of the sand. In the coarse aggregate, tough fresh quartzite particles are the most abundant constituent of this group. Quartz is a consistent minor constituent of the coarse and the fine aggregate. Chert composed of microcrystalline quartz is present in the gravel. Minor amounts of chalcedony were found in the sand. Pebbles of mylonite, granite, and gabbro are present in the coarse aggregate. In the sand, quartzite particles, clay, calcite, ore minerals, and accessory minerals such as zircon were assigned to this group.

APPENDIX B

LIST OF ROCK AND MINERAL NAMES FOR USE BY CONCRETE LABORATORY PETROGRAPHERS.[a]

ROCK NAMES

Group Terms:

1. Igneous Rocks
2. Volcanic Rocks
3. Basic Igneous Rocks
4. Acid Igneous Rocks
5. Basic Volcanic Rocks
6. Acid Volcanic Rocks
7. Glassy Volcanic Rocks
8. Porphyry
9. Metamorphic Rocks
10. Sedimentary Rocks
11. Carbonate Rocks

Major Rock Types:

1. Granite
2. Granodiorite
3. Diorite
4. Gabbro
5. Basalt
6. Rhyolite } Felsite
7. Andesite }
8. Pumice

9. Gneiss
10. Schist
11. Marble
12. Quartzite

13. Limestone
14. Shale
15. Slate
16. Siltstone
17. Chert
18. Sandstone
19. Graywacke
20. Conglomerate

Varietal or Minor Rock Names:

1. Syenite
2. Pegmatite
3. Gabbro-diorite
4. Amphibolite
5. Diabase
6. Serpentine
7. Dacite

8. (-) porphyry
9. Glassy (-)
10. Gneissic (-)

11. Argillaceous limestone
12. Arenaceous limestone
13. Cherty limestone
14. Shaly limestone
15. Oolitic limestone
16. Siliceous limestone
17. Magnesian limestone
18. Dolomitic limestone
19. Lithographic limestone
20. Chalk
21. Mudstone
22. Argillite
23. Phyllite
24. Argillaceous sandstone
25. Calcareous sandstone
26. Arkose
27. Arkosic quartzite

28. Coal
29. Lignite

30. Clay-ironstone

MINERAL NAMES

Group Terms:

1. Accessory minerals
2. Carbonate minerals
3. Clay minerals
4. Secondary minerals
5. Ferromagnesian minerals
6. Volcanic glass (a rock constituent but not a mineral)
7. Feldspar
8. Mica
9. Limonite
10. Wad
11. Amphibole
12. Pyroxene
13. Zeolite
14. Sulfides

Major Minerals (and Rock Components):

1. Quartz
2. Chalcedony (a mixture of a mineral and a mineraloid)

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[a] The terms given in this list have been used in petrographic reports issued by the Concrete Research Division, Waterways Experiment Station, or occur in published literature on concrete aggregates.

3. Opal (a mineraloid)
4. Tridymite
5. Cristobalite

Feldspar

6. Orthoclase
7. Plagioclase
8. Microcline

Pyroxene

9. Augite

Amphibole

10. Hornblende

Mica

11. Muscovite
12. Biotite
13. Chlorite
14. Calcite
15. Dolomite
16. Pyrite
17. Hematite
18. Magnetite
19. Gypsum
20. Kaolin

Volcanic Glass (not a mineral)

21. Acid Volcanic Glass
22. Basic Volcanic Glass
23. Intermediate Volcanic Glass

Varietal Terms and Less-Frequent Minerals

1. Gold
2. Graphite
3. Zircon
4. Garnet
5. Apatite
6. Allanite
7. Sphene
8. Epidote
9. Fluorite
10. Tourmaline
11. Galena
12. Pyrrhotite
13. Marcasite
14. Sphalerite
15. Chalcopyrite
16. Analcite
17. Natrolite
18. Laumontite
19. Nepheline
20. Sodalite
21. Olivine
22. Montmorillonite
23. Beidellite
24. Halloysite
25. Serpentine
26. Alunite
27. Bytownite
28. Andesine
29. Labradorite
30. Albite
31. Anorthite
32. Perthite
33. Microperthite
34. Aragonite
35. Siderite
36. Barite
37. Glauconite

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DISCUSSION

MR. H. S. MEISSNER.[1]—I should like to make a few comments in regard to allowing the petrographer to state his general opinion on the quality of the material. It has been my observation of the work of petrographers in the Bureau of Reclamation that, when they are skilled and have had considerable experience in observing the performance of aggregates in concrete under freezing and thawing and other durability tests, they then can predict very well what might be the performance of a new, unfamiliar aggregate.

Time and time again I have been impressed with their ability to predict what will happen, let us say, in the sodium-sulfate test, or even in freezing and thawing. Of course, I do not believe that I should extend such confidence to one who has not had a great deal of experience.

MR. BRYANT MATHER (*author*).—Our petrographers likewise have an extremely good record for their predictions concerning the behavior of materials which they have examined, but which have not yet been subjected to the laboratory freezing and thawing, magnesium-sulfate, or other tests. In our organization such predictions are regularly made. The point that I was trying to make earlier however, had to do with opinions as to the *suitability* of a material for use in field construction. We have gotten our fingers burned a little in connection with the examination of materials from regions with which we were unfamiliar. In such cases we have tended sometimes to jump to the conclusion that none of the samples submitted is fit for use in concrete. Well, they should not be, if there were better materials available, or if the structures to be built were such that they could not be repaired, or if the exposure were different. The petrographers have occasionally gone on record, informally, as to the entire unsuitability of a given material, which was subsequently used in the work with satisfactory results. The reverse is likewise true. After working for some time with materials of generally inferior quality, they then come across something that is a little better and it looks wonderful, but it comes from a region where there is lots of good material, so we do not use it. Since materials that are regarded by the petrographer as inferior often must be used when no better materials are economically available, and since certain materials of good quality often are not used because they are not available in sufficient quantity, are located at too great a distance, or because even better materials are available; it is my feeling that remarks about suitability for field use should be left out of formal petrographic reports. I do believe however, that a petrographer should give his speculations and predictions the fullest expression within the organization in which he works; the making and confirming of such predictions will add to the confidence of the petrographer and to his worth to the organization.

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MR. C. H. SCHOLER.[2]—I should like to ask how fast a man can work. That is, if there are 300 particles of various types, quite a problem is entailed in making a study. How soon can they get results to the engineer?

MR. MATHER.—I believe it is brought out in the paper that we contemplate two major types of petrographic examination, a preliminary examination and a complete examination. We have made preliminary examinations while the engineer has held the 'phone. The precision of the quantitative information, if any, provided by such an examination is quite low. You can do any amount of work, depending merely upon time and funds, on any sample.

We feel that for a complete examination of an aggregate to be used in important work, the precision of the quantitative results requires the examination of the quantities of material indicated.

Specifically, the time required to sort out 300 particles varies with the heterogeneity of the group of particles considered. It varies anywhere from ten minutes to half-a-day. If, as is the case with a great many samples, you have only two or three constituents which can be identified by inspection, the work goes very rapidly. With the apparatus shown in Figs. 1 and 6 of the paper, you can have one hand on the microscope and the other hand on the counter, and you can click the particles off just about as fast as you can roll them past your field of vision. In other cases, it is necessary to pick out particles for more detailed determination of their properties before they can be tallied.

We make a quick, preliminary examination of a material of relatively simple composition in a half-day, by one man, including the notes for the report. If the composition is complex, it takes one man a full day or a day-and-a-half. The more detailed examination takes several days.

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PULSE TECHNIQUES APPLIED TO DYNAMIC TESTING*

By J. R. Leslie[1]

Synopsis

Many of the difficulties inherent in present methods of dynamic testing can be overcome if pulse techniques are utilized. In this method the velocity of propagation of an acoustical pulse through the material is measured. The pulse velocity so obtained is unaffected by size or shape of the specimen, and the elastic modulus may be calculated using one simple formula for all cases. An electronic pulse test set developed primarily for testing concrete structures in the field is described. A pulse generator and receiver are used, applied to opposite faces of the structure. The time of travel of the pulse is measured on a cathode ray tube using a strobe circuit and direct reading dial. More than two years' experience in testing laboratory specimens, for which it is now in regular use, is described. Mass concrete in the field has been tested for both deterioration and cracking; in one case many thousand readings were taken. The instrument's application to the location of rot in wood pole specimens and to the testing of soils is also illustrated.

Many of the difficulties inherent in the present methods of dynamic testing can be overcome if pulse techniques are utilized. In this method, the velocity of propagation of an acoustical pulse through the material is measured, whereas in the sonic method in common use in concrete laboratories the resonant frequency is obtained in flexural or torsional vibration. In contrast to the latter method, the pulse velocity is unaffected by size or shape of the specimen, so that non-destructive tests may be performed on laboratory or field specimens. The elastic modulus may be calculated from the information so obtained, using one simple formula for all cases.

Pulse techniques were first widely used in the recent World War, probably the first engineering application being in the underwater detection of submarines. The most widely known peacetime application is the ultrasonic flaw detector developed for the purpose of locating cracks in metals. All of these methods depend on the measurement of the time of travel of a pulse through the medium to the object or flaw and back to the transmitter after reflection. The velocity of propagation is assumed to be a known quantity.

The dynamic testing method described in this paper depends on a measurement of the time of travel—or pulse velocity as it will be called—from one face of the body to the opposite face. Several scientific papers have described the use of pulse techniques in chemical (**1**),[2] physical (**2**), and other (**3**) fields. Very recently its use in engineering applications has been revealed. The author, in an earlier paper (**4**), described the use of a pulse test set in the examination of dams for cracking. As a result of this work, the possibilities with regard to dynamic testing of materials were realized. Work of a similar nature has been reported independently by the Road Research Laboratory in England (5). The present paper outlines the theory and the use of pulse techniques, describes the design features of a test set for this purpose, and gives the results of more than two years' experience with it in concrete deterioration studies. This experience has enabled the condition of concrete to be determined from the pulse velocity alone. It appears that for standard concretes pulse velocities of 13,000 to 16,000 ft. per sec. indicate that the condition of the concrete is good to excellent. Velocities under 10,000 ft. per sec. indicate concrete in poor condition.

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Research Engineer, The Hydro-Electric Power Commission of Ontario, Toronto, Ontario, Canada.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1323.

The Soniscope, as the instrument is called, is now in regular use by The Hydro-Electric Power Commission of Ontario for locating deterioration in concrete in the field, in long-time studies in changes of concrete, and in exploring the depth and extent of cracks in dams. In an extensive survey of one dam constructed in 1914, more than 5000 pulse velocity readings have been taken.

THEORY OF PULSE TESTING

The theory of the transmission of impulses through a solid body was first examined in connection with the propagation of earthquake waves through the earth. Any impulse generated in an extended solid can be shown to separate into three groups of waves, a compressional, a shear and a surface wave, each travelling with a different velocity. They are sometimes termed longitudinal, transverse, and Rayleigh waves, respectively. Each travels with a velocity determined by the elastic constants and the density of the medium. The compressional waves travel with the greatest velocity and are the chief concern of the present paper. They arrive at the receiver prior to the other two waves and are not interfered with by them. The arrival time can be determined accurately, and from the resulting velocity the dynamic elastic modulus is simply calculated using the following equation (4):

$$E = dV^2 \frac{(1+\mu)(1-2\mu)}{(1-\mu)} \quad \ldots\ldots (1)$$

where:

E = dynamic modulus of elasticity,

d = density,

V = compressional pulse velocity, and

μ = Poisson's ratio.

Because variations in μ affect the result to only a minor degree, the last term may be termed constant for a particular material, to a first approximation. Thus the equation becomes:

$$E = CdV^2 \quad \ldots\ldots (2)$$

The pulse velocity so measured is the true group velocity in the medium, and it may differ from the wave velocity as measured in the sonic method. Group velocities are independent of boundaries in the medium provided reflections from them do not arrive at the receiver in time to interfere with the leading edge of the directly transmitted pulse.

DESCRIPTION OF PULSE TEST SET

General Procedure:

The apparatus, termed the Soniscope (Fig. 1), sends pulses of high-frequency sound into the material and measures the time of travel from the transmitter on one face to the receiver on a distant face, by electronic means.

Simultaneously with the transmission of the pulse, the beam of a cathode ray tube is made to sweep linearily across the face of the tube. The arrival of the pulse at the receiver is recorded as a series of vertical oscillations of the trace.

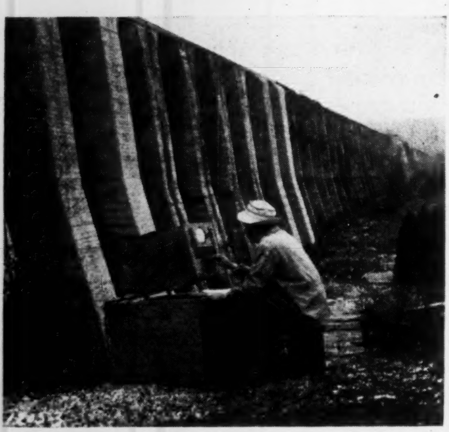
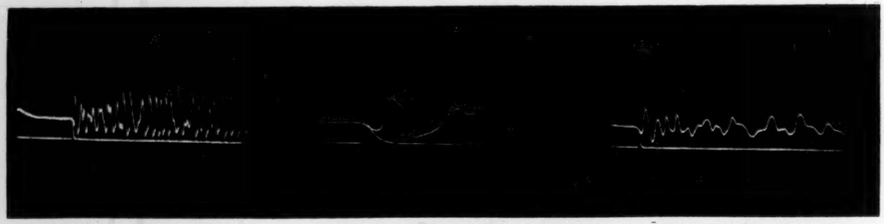


Fig. 1.—Field Testing of Mass Concrete with the Soniscope.



(a) Transmission Through 2.5 ft. of Concrete.

(b) Expanded View of Received Pulse in (a).

(c) Transmission Through 4-in. Soil Specimen.

Fig. 2.—Typical Records.

The elapsed time is therefore indicated on the face of the tube by the position of the wave front of the received oscillations. This time interval is accurately measured by means of a calibrated marker pulse or strobe on a second trace of the cathode ray tube. The strobe is aligned opposite the leading edge of the received pulse, its position being controlled by a graduated potentiometer directly calibrated in microseconds.

Typical photographs of the cathode ray tube face are reproduced in Fig. 2. On the upper trace of each photograph the transmitted pulse is represented by the left-hand edge of the trace. The received oscillations show the wave front of the pulse followed by resonant oscillations of the crystals. The timing strobe is visible on the lower trace, set opposite the leading edge of the received pulse. Figure 2(*a*) shows transmission through 30 in. of concrete, in this case a 6-in. core removed from a dam. Figure 2(*b*) shows an expanded view of the first received cycles in Fig. 2(*a*). The expansion is performed electronically and serves to increase the accuracy of setting the strobe. The time interval in the core was measured as 182 microseconds, giving a velocity of 13,700 ft. per sec.

Figure 2(*c*) shows the transmission through a 4-in. specimen of soil.

The procedure is to mark two positions on a structure a known distance apart. After wetting these areas with water, the transducers are applied under slight pressure. The operator then measures the elapsed time, enabling the velocity to be calculated. The operator is required to operate a minimum of controls and switches; so the procedure can be learned by a technician with little difficulty. Two assistants are, of course, required to hold the transmitter and receiver against the body under test. In the case of routine tests in the laboratory, they have been replaced by a jig which holds the units against the specimens.

Pulse Transmitter Design:

The pulse is generated by means of piezoelectric crystals of Rochelle salt. When subject to the discharge voltage from a condenser charged to a few hundred volts, the crystals vibrate mechanically for a few cycles at their resonant frequency. With the crystals pressed against the solid under test, the acoustical pulse is transmitted into the solid.

Transmission of the pulse into the material was found to be greatly decreased by the presence of an air gap, however small, between the crystal and specimen, the loss in energy taking the form of reflection of the pulse at the crystal-air interface and again at the air-solid boundary. The reflection loss is decreased by the provision of a liquid film between the crystal and the solid. Polishing the face of the material, by decreasing the film thickness, results in a further decrease in loss.

Early experimental apparatus used unmounted crystals of 45-deg. X-cut Rochelle salt 1 by $1\frac{1}{2}$ by $\frac{1}{4}$ in. in size pressed against the faces of the solid with a film of petrolatum or glycerine in the boundary; however, this procedure was not suitable for routine tests. The pulse generator finally evolved (Fig. 3) consists of an assembly of crystals immersed in an oil filled brass housing.

The housing is provided with a thin rubber window directly in front of the crystal face, through which the sound pulses pass. By providing slight excess pressure inside the housing, the rubber window takes up a convex shape. Pressure of the housing against the material under test results in the rubber flattening against the specimen, the latter not requiring in this case a polished surface. The surface may nevertheless require wetting by water or petrolatum in order to exclude minute amounts of air trapped in the surface.

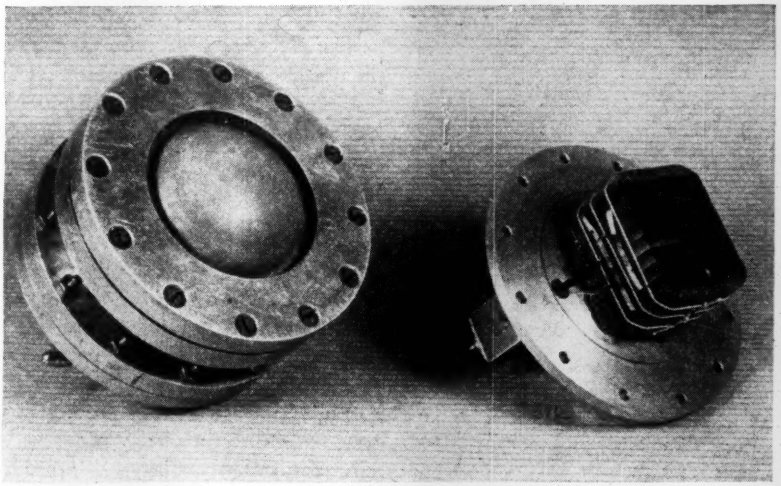


FIG. 3.—Pulse Transmitter.

The slope of the leading edge of the received pulse determines the accuracy with which the elapsed time can be measured. A pulse frequency of 20,000 cps. allows the wave front to be determined within a few microseconds, or to an accuracy of about 2 per cent in a 20-in. specimen of concrete. Pulse frequencies higher than 100,000 cps. have been used (5) with corresponding increase in accuracy, but at the expense of a decrease in range through the material. The range of the higher frequency pulses is limited due to the increase in attenuation with frequency. Thus, in concrete, use of the high frequencies is limited to specimens a foot or so in length (or to 1-ft. path lengths in larger bodies). Frequencies of 20,000 cps. have been successfully used in mass concrete up to 50 ft. in thickness. Attempts to increase this range by enlarging the transmitter have not been successful up to the present time.

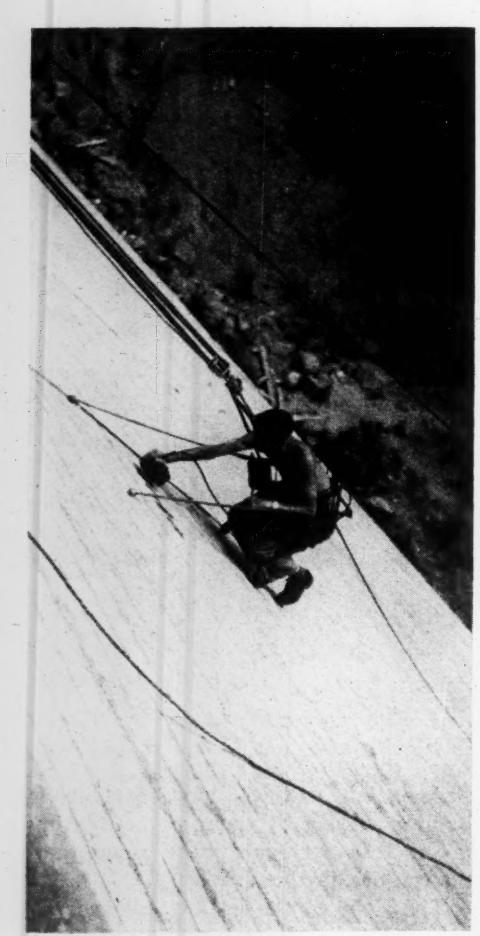


FIG. 4.—Pulse Receiver Testing Mass Concrete.

The range depends on the background noise level. It is considerably reduced in the vicinity of heavy construction, riveting, etc.

The receiver is similar in construction to the transmitter. A small pre-amplifier is mounted with it, the latter terminating in a low impedance output for connection to a coaxial cable. It is shown in Fig. 4 in use on a concrete structure.

Electronic Circuits:

The electronic circuits, although complicated, are for the most part an assembly of circuits well known to specialists in the art. Only a few of the circuits will be treated in detail. A block diagram is shown in Fig. 5.

The multivibrator controls the operation. It produces square waves at a repetitive rate of 100 per second. The output is differentiated to obtain positive and negative trigger pulses which initiate the pulse generator, sweep, and timing circuits.

The pulse generator circuit is shown schematically in Fig. 6. The multivibrator trigger voltage is amplified by V_1, which is a miniature type thyratron. Condenser C_1, which has charged through R_1 to B+ voltage, discharges through the tube to produce a positive trigger of 300 v. amplitude. This trigger fires the second thyratron. Tube V_2 is hydrogen filled and requires a high trigger voltage. Its maximum ratings are 3000 v. on the plate and 30-amp. surge current. Condenser C_2 is thus discharged directly into a 50-ohm coaxial cable producing an 800-v. exciting pulse for the crystal transmitter. This pulse is limited in size only by the voltage rating of V_2 and C_2. The low impedance (50-ohm) output pulse is necessary if long cables are to be used between the control unit and the transmitter. Leads more than 300 ft. long have been used in examining large dams. In this instance telephone communication may be necessary between the operators.

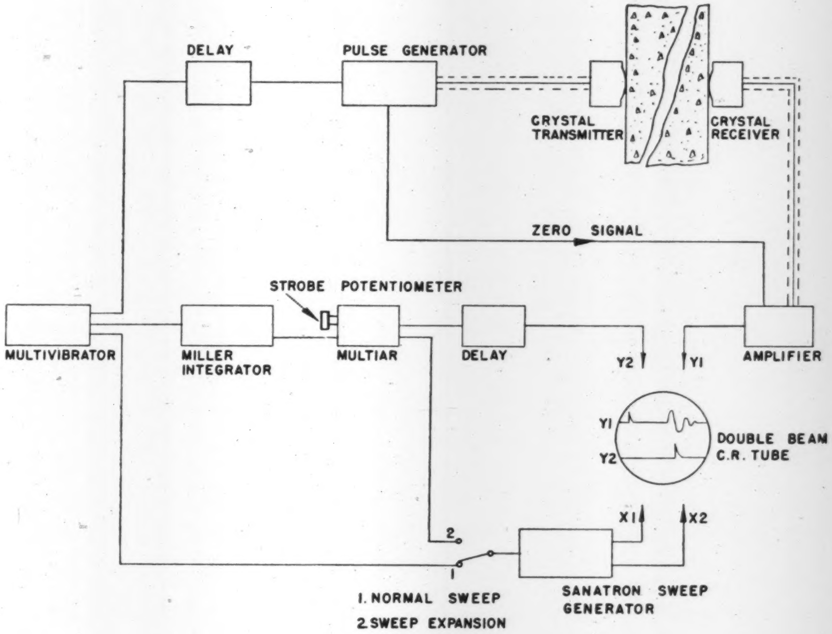

DELAY
PULSE GENERATOR
CRYSTAL TRANSMITTER
CRYSTAL RECEIVER
ZERO SIGNAL
STROBE POTENTIOMETER
MULTIVIBRATOR
MILLER INTEGRATOR
MULTIAR
DELAY
Y2
Y1
AMPLIFIER
Y1
Y2
DOUBLE BEAM C.R. TUBE
X1
X2
2
1
1. NORMAL SWEEP
2. SWEEP EXPANSION
SANATRON SWEEP GENERATOR

FIG. 5.—Block Diagram of Electronic Circuits.

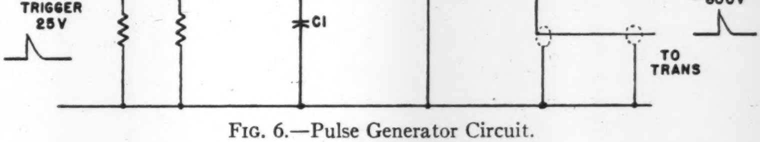

+300V
+800V
V1 = 2D21
V2 = 3C45
R1
R2
V1
V2
C1
C2
TRIGGER 25V
-800V
TO TRANS

FIG. 6.—Pulse Generator Circuit.

The trigger pulse serves a second function, that of initiating the sweep circuit connected to the horizontal deflection terminals of the cathode ray tube. The sweep circuit consists of a self-gated Miller integrator circuit termed a Sanatron (**6**, **7**). This circuit produces a linear saw-tooth voltage. Sweep times varying from 50 to 5000 microseconds are available by varying an *RC* time constant in the Miller integrator.

The trigger pulse also initiates a marker pulse or strobe, which is used to measure the time interval. This portion of the circuit presents the greatest difficulty to the designer. Several types of strobe circuits were developed during the war for radar use. The arrangement finally adopted consists of a Miller integrator in combination with a multiar pick-off circuit. It has been discussed in the literature (**6, 7**) and need not be described here. The potentiometer, which aligns the strobe opposite the wave front of the received pulse, can be directly calibrated in time to an accuracy of one part in five hundred. The calibration is little affected by changes in line voltage or temperature.

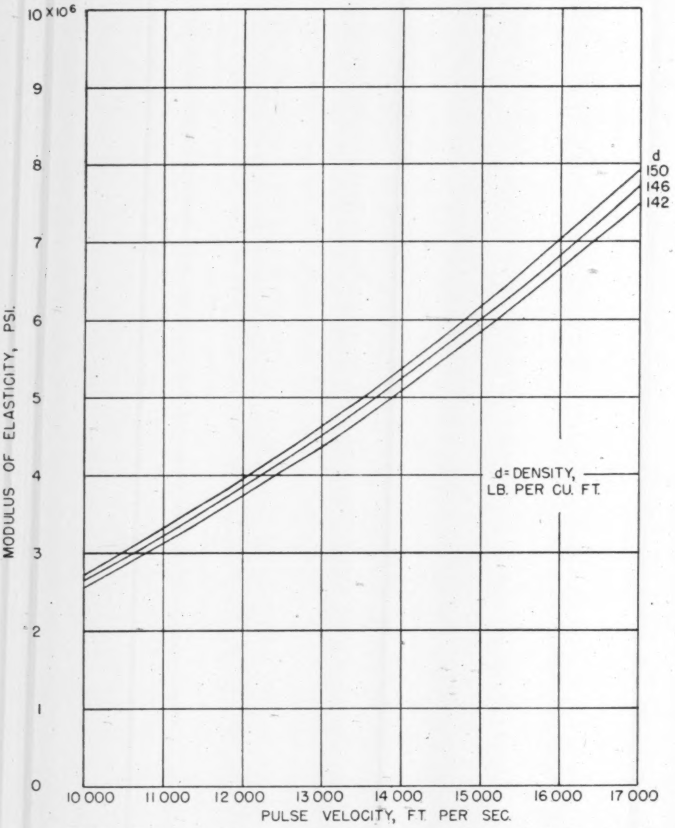


FIG. 7.—Relation Between Dynamic Modulus and Pulse Velocity for Concrete.

Fixed delays are inserted in the circuits to allow the sweep voltage action to commence before the thyratron pulse appears. Otherwise the latter pulse would appear at the extreme left-hand edge of the screen.

A sweep expansion circuit may be used to examine any portion of the trace in more detail. This is performed by a switching arrangement which allows the sweep generator to be triggered a few microseconds prior to the strobe. The switch also serves to speed up the sweep by a factor of five. Thus any portion of the normal sweep can be examined by setting the strobe opposite it, and switching to the sweep expansion position (Fig. 2(*b*)).

The control unit containing all the above circuits is shown in Fig. 1. It contains 32 tubes in all and a small blower fan for cooling purposes. The weight of the unit is 60 lb.

LABORATORY TESTS

Concrete:

Prior to the use of the Soniscope in the routine testing of laboratory and mass concrete, a check on the accuracy of Eq. 1 or 2 was required against standard test procedures. A number of 4 by $3\frac{1}{2}$ by 16-in. specimens of concrete were secured and the modulus obtained in accordance with the A.S.T.M. Tentative Method of Test for Fundamental Transverse Frequency of Concrete Specimens for Calculating Modulus of Elasticity (Sonic Method) (C 215 – 47 T).[3] The pulse modulus was then measured. The results, quoted in the earlier paper (4), showed a satisfactory correlation between the two methods.

A value for Poisson's ratio was also obtained from the sonic tests. These values were higher than values quoted, a figure of 0.24 being representative of mixtures of normal density.

The majority of test cylinders made up in our concrete laboratory in connection with proportioning tests and durability studies are now tested in routine fashion with the Soniscope. A simple graph (Fig. 7) is used to obtain a value for the modulus. The value of E so found is believed to be accurate to ±5 per cent.

Many miscellaneous tests have been performed during the past two years. They illustrate the versatility of the pulse method. The increase in strength of plastic concrete when setting is easily demonstrated by measuring pulse velocity. Expansion in mortar bars has also been correlated with the velocity. Concrete damaged in a fire on a construction job was also tested. The damage was found to be only superficial.

A number of precast slabs made up in the form of concrete-foam glass sandwiches were tested for bond strength under the action of temperature differentials between the faces of the sandwich. The pulse velocity served as a sensitive indication of the deterioration of the bond in the sandwich.

TABLE I.—PULSE VELOCITY TESTS ON WOOD.

Specimen	Condition of Specimen	Velocity Parallel to Grain, ft. per sec.	Velocity Perpendicular to Grain, ft. per sec.
No. 1......	Sound specimen as received................	14 700	5 400
	Sound specimen, dried in air one week..........	15 180	5 400
No. 2.......	Sound..................	13 200	5 820
No. 3.......	Some rot present (a borderline case)...........	12 600	5 300
No. 4.......	Rot present, a rejectable sample...............	12 000	4 300
No. 5......	Rot present, a rejectable sample................	11 600	3 400
	Rot present, dried in air one week..............	11 900	3 600
No. 6.......	No rot, specimen had a "shake" in the core....	12 700	4 300

Wood:

With the object of determining the presence of heart rot in wood poles, a short series of pulse velocity tests were made on Jack pine specimens (*Pinus banksiana*) with various amounts of heart rot present. The specimens were in all cases about 3 ft. long and 10 in. in diameter. Velocities measured parallel and perpendicular to the grain are shown in Table I.

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[3] 1949 Book of A.S.T.M. Standards, Part 3, p. 855.

Because of the limited nature of the tests, few conclusions can be safely drawn; however, they do show a correlation between the pulse velocity and the condition of the wood. The velocity is dependent also on moisture content and probably other factors. The elastic modulus can be calculated if a proper value of Poisson's ratio is used. The method may be a valuable research tool in the comparison of wood samples, but this possibility has not been explored.

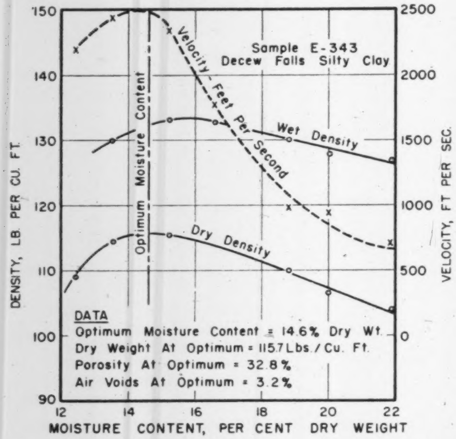

FIG. 8.—Velocity in Silty Clay with Different Moisture Contents and Compactions.

Soils:

Limited experiments of a preliminary nature were carried out on soils. One sample was silty clay. Using a standard Proctor compaction mold for compacting the sample, the pulse velocity was first measured for varying densities. The velocity was found to be a maximum as the percentage of moisture reached its optimum value (Fig. 8). Compaction was then varied with the soil at optimum moisture content, and velocities obtained. The velocity was found to be proportional to the dry density under these conditions (Fig. 9). Thus, with the use of control tests in the laboratory, the density may be obtained directly in the field with a minimum of effort.

FIELD TESTS

The principal application of the method in the field has been to the examination of mass concrete in dams. Subject to a limitation in range of 50 ft., any structure in the field may be so tested. Because the transmitter and receiver are relatively nondirectional, transmission times may be measured across the corners of a section, or they may be measured along the downstream surface of the dam. In the latter case the modulus of the surface layer only is of course obtained. Pavement slabs may be similarly treated, the transmitter being held against the shoulder of the slab.

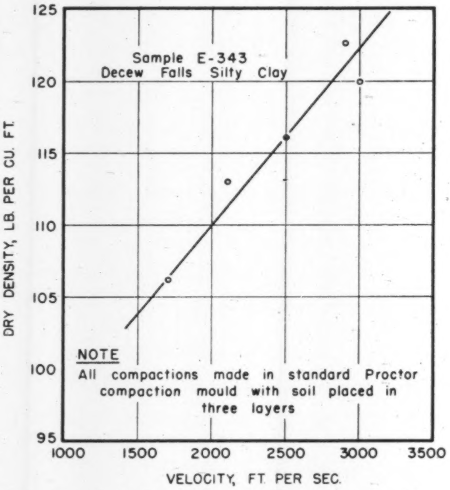

FIG. 9.—Relation Between Velocity and Dry Density in Silty Clay.

A thorough inspection is being made of one of the older dams owned by the Commission. This dam was constructed in 1914 and has required repairs three times since that date. The dam is of the slab and buttress type and is shown in Fig. 1. The slab is of reinforced concrete 12 in. thick at the top, increasing to a maximum of 30 in. in the deepest sections.

To test the concrete in the slabs of this dam, the transmitter was lowered into the water on the upstream side and the receiver held opposite it on the downstream side. Areas judged to be in poor condition had previously been located by tapping with a hammer and outlined with paint. All concrete inside the areas outlined was found to show velocities of less than 10,000 ft. per sec. and only two points were above 5000 ft. per sec. The significance of these low readings can perhaps be better appreciated by considering that since the normal velocities th ough adjacent areas were over 15,000 ft. per sec., the poor areas have a modulus of only about 10 per cent of that found in good areas.

Sixty-three slabs have to date been tested and the work is proceeding. More than 5000 readings have been taken In general the low velocity areas agree fairly well with those located using a hammer, but in all cases considerably larger areas of partial deterioration were indicated by the Soniscope.

Another concrete hydraulic structure tested with the Soniscope is a surge tank approximately 60 ft. square and 30 ft. high, with walls about 8 ft. thick. This structure was built in 1904 and has been in continuous use since, in a very rigorous climate. Considerable spalling has taken place on the south and west outside surfaces, that is, those probably subjected to the greatest number of freezing-and-thawing cycles. The Soniscope tests revealed that the concrete in this structure is generally in excellent condition, having a pulse velocity of 15,000 to 16,000 ft. per sec. The deterioration which produced the spalling was found to be only 1 ft. in depth at the greatest and on the average was only 1 or 2 in. deep.

Soniscope tests were carried out on one portion of a dam during construction. Control cylinders for three sections of this structure showed evidence of low strength. These sections were subjected to pulse tests. The velocities were found to be of the order of 11,000 ft. per sec. whereas other sections showed values of 13,000 ft. per sec. The field check thus confirmed the routine control procedures

Three pulse test sets are now in use by the Commission. One is in the Concrete Laboratory with the others available for field testing.

CONCLUSIONS

Non-destructive testing in many solids have been shown to be facilitated by the use of pulse techniques. The pulse velocity is unaffected by the size or shape of the specimen and may be used to calculate the dynamic modulus of elasticity using one formula for all cases.

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(3) H. B. Huntington, A. G. Emslie, and V. W. Hughes, "Ultrasonic Delay Lines," *Journal of the Franklin Institute*, Vol. 245, Nos. 1 and 2, Jan. and Feb. 1948, pp. 1 and 101.

(4) J. R. Leslie, and W. J. Cheesman, "An Ultrasonic Method of Studying Deterioration and Cracking in Concrete Structures," *Journal*, Am. Concrete Inst., Vol. 21, No. 1, September, 1949; *Proceedings*, Vol. 46, p. 17–37.

(5) R. Jones, "The Non-Destructive Testing of Concrete," *Magazine of Concrete Research* (London), No. 2, June, 1949, p. 67.

(6) F. C. Williams and N. F. Moody, "Ranging Circuits, Linear Time-Base Generators and Associated Circuits," *Journal*, Inst. Electrical Engrs., (London), Vol. 93, Part IIIA, No. 7, March–May, 1946, p. 1188.

(7) "Waveforms," M. I. T. Radiation Laboratory Series, Vol. 19, Chapters 5 and 9, McGraw-Hill Book Co., Inc., New York, N. Y. (1949).

DISCUSSION

MR. C. H. SCHOLER.[1]—I wish to compliment Mr. Leslie in being a pioneer in the use of what I think promises to be a very fruitful and remarkable tool for studying engineering structures and parts of engineering structures.

We started the use of this type of equipment about a year ago when we constructed an experimental concrete pavement. A portion of this pavement was built using materials which we were confident would deteriorate within a very few years, and other portions in which we believed effective measures had been taken to prevent development of this type of deterioration.

In studying the characteristics of the concrete which we placed, we began taking readings of the apparent velocity of sound in the concrete just as the material was changing from a plastic to a rigid material. Readings could be secured in about 3 hr. Just as the change started to take place, probably initial set, the velocity changed rather quickly from slightly below the velocity of sound in water to higher velocities, increasing to 8000 to 10,000 ft. per sec., within a matter of 24 hr., and since then, up to 6 months, a gradual and consistent increase in velocity has occurred.

These readings will be taken continuously until deterioration develops in the inferior sections. It is hoped that this tool may be a satisfactory measure of the degree of deterioration that results.

I am also much interested in the possible application of such techniques to the study of subgrade soils which are supporting pavements of various types. Our studies of existing paving structures have been particularly weak in that we have no non-destructive method of measuring changes in the rigidity and bearing power of the soil underneath our pavements. If such a technique can be applied to these problems, it should be invaluable in evaluating the effects of loads on rigid and flexible pavements.

MR. H. E. DAVIS.[2]—I should like to ask a question of Mr. Leslie. In connection with the tests on compacted soils, were you able to detect differences in moisture content of soils compacted to the same density, or were all these tests made on soils in the dry condition?

MR. J. R. LESLIE (*author*).—We performed two series of tests, one with various moisture contents and the other with various compactions and the tests illustrated were at the optimum moisture content for the soil, but with varying compactions. The velocity was found to increase with compaction.

With constant compaction the velocity varied with the moisture content and was an optimum with the optimum moisture content in the soil. The practical difficulty is the simultaneous variation in the field of both these variables.

MR. H. S. MEISSNER.[3]—The results you show in Fig. 8 are very interesting since they show decided relations between velocity and soil density. It suggests that this apparatus may be found to be a useful tool in earthwork control.

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[1] Head, Dept. of Applied Mechanics, Kansas State College, Manhattan, Kans.

[2] Director, Institute of Transportation and Traffic Engineering, University of California, Berkeley, Calif.

[3] Bureau of Reclamation, Denver Federal Center, Denver, Colo.

MR. G. C. WILSNACK.[4]—I wonder whether Mr. Leslie has experimented with this apparatus in connection with geological work to locate depths and nature of rock in the field.

MR. LESLIE.—Some work of this nature has been carried out; however the instrument is very sensitive to cracks in the solid, because it was designed originally to detect cracks in dams. It will detect cracks too small to be visible.

We did make some tests through bedrock outcroppings, but the transmission was very poor, and I think the maximum range was 5 or 10 ft.

At that time it was apparent that minute cracks existed in the bedrock in every direction even though the latter was thought to be solid.

In addition, the rock changed in density and in grain structure, resulting in increased absorption of the sound beam. I believe that transmission through rock is only possible using very low-frequency waves.

MR. I. L. TYLER.[5]—With considerable assistance from Mr. Leslie and his associates, we have built two of these machines, and we have two more under construction.

We believe that this test offers some real opportunities for studying the performance of concrete that cannot be realized by any other means of which we are aware. We have a good opportunity to check the value of the test in connection with the specimens of the Long-Time Study of Cement Performance in Concrete. There is one project in which there are concrete mixes that are almost identical, except for the coarse aggregate. There is a range with three types of coarse aggregate of about 3000 ft. per sec. in what we believe to be concretes of comparable quality. With one aggregate the velocity is around 16,000 and with another it is around 13,000. The third is intermediate, about half way between those two. So it is not quite the simple operation of taking the equipment out, leaning it up against a concrete structure and coming away with a perfect picture of what goes on inside. We think it has wonderful possibilities, but we believe that considerable study will be necessary to put it into really dependable use.

It is suggested that if anyone has a notion of building this new equipment he does not attempt it in his spare time. It is an extremely complicated affair.

MR. BRYANT MATHER.[6]—To pick up where Mr. Tyler left off, I wonder if Mr. Leslie has any suggestions about how an organization might best go about getting a soniscope.

MR. LESLIE.—The Hydro-Electric Power Commission has been making arrangements to have these instruments built more or less to order by a company in Toronto. This company is very expert in the electronic field, they do a lot of research for several large organizations, and I think they are quite competent. I would be pleased to put anyone, interested in purchasing an instrument of this type, in touch with this firm.

MR. MEISSNER.—I should like to ask Mr. Leslie another question: He referred to the application of this test to timber. I did not quite gather whether that was new timber, before it had gone in place, or was it power-line transmission poles in place, in the ground?

MR. LESLIE.—The samples were new specimens of jackpine which had been obtained from our storage yard and were regarded as representing both sound wood and also specimens that had heart rot in them. A typical problem would be to differentiate between 3000 or 4000 poles, in a storage yard—whether we could separate them into good and bad lots.

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[4] Ideal Cement Co., La Porte, Colo.

[5] Manager Field Research, Portland Cement Assn., Chicago, Ill.

[6] Engineer, Concrete Research Div., Waterways Experiment Station, Jackson, Miss.

We took about five samples, and there did seem to be a correlation; however this work was not pursued further.

We have thought of using the method to check transmission line poles in place, but it is rather expensive, and time has not been available. Some companies have tried to X-ray poles, which is an even more expensive proposition.

MR. F. J. MARDULIER.[7]—I should like to ask Mr. Leslie what effect the moisture content in the concrete has on the pulse velocity.

MR. LESLIE.—For many years now, an extensive series of tests regarding the effect of moisture content on velocity has been planned, but these have not been carried out to date. We did take a few specimens and having immersed them for awhile and then dried them out in the air, obtained the velocity under each condition. It changed a few per cent only.

The limits in accuracy of the instrument at the moment are about 1 or 2 per cent, consequently no definite conclusions were reached.

MR. TYLER.—I might add a little to that. With the cooperation of R. E. Davis at the University of California, we had a chance to make a few tests on some laboratory-cured specimens, part of which had been cured approximately 28 days in a moisture room. The other portion had been placed under alternate vacuum and water pressure to saturate the specimens as completely as possible. The reductions in velocity were of the order of 1000 ft. per sec. on specimens which before saturation had velocities of 16,000 ft. per sec.

This was an exaggerated condition. It is very doubtful that the same degree of saturation could be produced by ordinary exposure.

MR. E. P. PITMAN.[8]—Can this test be applied to routine testing, say, 28 days' strength, or is it only for deterioration of concrete at older ages.

MR. LESLIE.—The instrument essentially measures the dynamic modulus to an accuracy of, say, 2 or 3 per cent, and it will do everything that the sonic tests will do. In addition measurements can be made in the field, 28 day tests being perfectly feasible with the soniscope.

MR. R. R. LITEHISER.[9]—I could not determine from Mr. Tyler's remarks whether velocity increased or decreased with the increase in water.

MR. TYLER.—The velocity decreased with more water.

MR. LESLIE (*author's closure*).—In a more recent series of tests on the effect of moisture content on pulse velocity, twenty-six 6 by 12-in. cores removed from a dam 5 yr. ago and stored in air since that time, were measured with the soniscope. The cores were then immersed in water at room temperature until they reached constant weight. The pulse velocity was again measured and found to average 12 per cent greater than before. Maximum increase was 20 per cent, minimum 6 per cent.

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[7] Cement Div., Dewey and Almy Chemical Co., Cambridge, Mass.

[8] The Port of New York Authority, New York, N. Y.

[9] Chief Engineer, Testing and Research Lab., Department of Highways, Columbus, Ohio.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON USE OF POZZOLANIC MATERIALS IN MORTARS AND CONCRETES

This Symposium on Use of Pozzolanic Materials in Mortars and Concretes was held at the Cement and Concrete Session of the First Pacific Area National Meeting of the American Society for Testing Materials in San Francisco, Calif., October 10–14, 1949.

The following nine papers were presented:

Introduction—T. E. Stanton
Pozzolanic Materials and Their Use in Concrete—R. E. Davis
Pozzolans Used in Mass Concrete—H. S. Meissner
Experience with Pozzolanic Materials in Kansas—C. H. Scholer and R. L. Peyton
Effect of Calcination on Natural Pozzolans—R. C. Mielenz, L. P. Witte, and O. J. Glantz
Effect of the Use of Diatomite Treated with Air-Entraining Agents upon the Properties of Concrete—R. E. Davis and A. Klein
Summary of Methods for Determining Pozzolanic Activity—W. T. Moran and J. L. Gilliland
Strength, Volume Change and Sulfate Resistance of Mortars Containing Portland-Pozzolan Cements—R. E. Davis, W. C. Hanna, and E. H. Brown
Studies of Some Methods of Avoiding Expansion and Pattern Cracking Associated with the Alkali-Aggregate Reaction—W. C. Lerch
Studies of Use of Pozzolans for Counteracting Excessive Concrete Expansion Resulting from Reaction Between Aggregates and Alkalies in Cement—T. E. Stanton

These papers and discussions are issued as A.S.T.M. Special Technical Publication No. 99, entitled "Symposium on Use of Pozzolanic Materials in Mortars and Concretes."

PILOT STUDIES OF SOIL DENSITY MEASUREMENTS BY MEANS OF X-RAYS*

By D. Berdan[1] and R. K. Bernhard[1]

Synopsis

Pilot experiments have been made with soil samples in small containers to determine, by means of X-rays, first, the effect of soil densification on the radiographs and, second, the effect of densification on differential pressure cells buried in the soil samples. Characteristic X-ray images are presented, and some conclusions with respect to the application of this method are discussed.

This nondestructive X-ray method leaves the soil sample completely unchanged, regardless of its state of densification, and might become useful particularly in those cases where undisturbed soil samples cannot be obtained. A possible development for future field applications is suggested.

Pilot experiments with soil samples in small containers were made to determine the effect of soil densification on radiographs, and the corresponding motion of differential pressure cells buried in the soil. Since it is often difficult to obtain completely undisturbed soil samples, particularly in the case of noncohesive dry sands, the X-ray method appeared to offer a promising approach (**1**).[2] To avoid misunderstanding, it will be understood that "soil density" refers to densifying effects due to compaction of soil, and "radiograph or print density" refers to darkening effects due to the exposure of radiograph film or print.

Test Procedure

The general setup for the experiments is shown in Fig. 1.

X-ray Unit:

The X-ray machine (Westinghouse Industrial Unit) is a two-valve, full wave rectification unit (**2**) with a maximum rating of 150 kv. An automatic timer governs the exposure time. Images were taken before and after densification of the soil samples.

Soils:

Two soil types of widely contrasting composition were selected: first, an Atlantic Coast beach sand (noncohesive) and, second, a Hagerstown Silt Soil (cohesive). In Fig. 2, their grain size accumulation curves are represented. The sand consisted of 95 per cent quartz and 5 per cent feldspar; the Hagerstown silt soil contained approximately 15 per cent quartz and 85 per cent mica, clay, minerals and iron oxides. Further constants of the Hagerstown silt soil are: liquid limit 38.5 per cent, plastic limit 22.1 per cent, and plasticity index

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* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

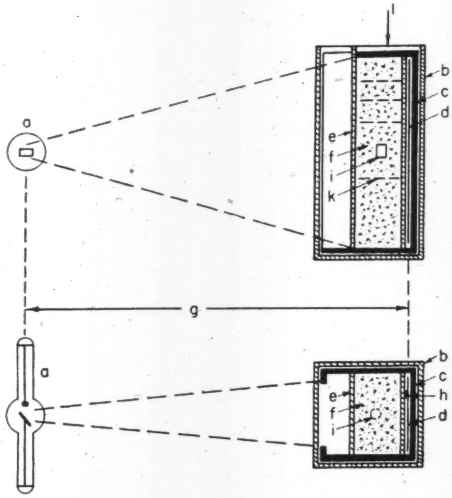
[1] Senior Student of Mechanical Engineering and Professor of Engineering Mechanics, Rutgers University, New Brunswick, N. J.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1337.

16.4 per cent. Both soils were tested in dry condition.

Container:

The soil was poured as loosely as possible into a wooden container (*b*, Fig. 1), which was 22 in. high and had a cross-section of 9 by 9 in. (inside dimensions). Lead shields *c* masked all sides of this container, with the exception of the front, to reduce scattered radiation.[3] The thickness of the soil sample could be varied by inserting an adjustable separation wall *e*. An X-ray film holder, *d*, fitted into the back of the container, and a scale, *h*, consisting of lead markers attached to a wooden strip, was placed in front of the film.



(*a*) X-ray tube
(*b*) Wooden container
(*c*) Lead shields
(*d*) X-ray film
(*e*) Adjustable separation wall
(*f*) Soil sample
(*g*) Distance: focal spot-X-ray tube to film
(*h*) Vertical scale
(*i*) Differential pressure cell
(*k*) Aluminum foils
(*l*) Applied load.

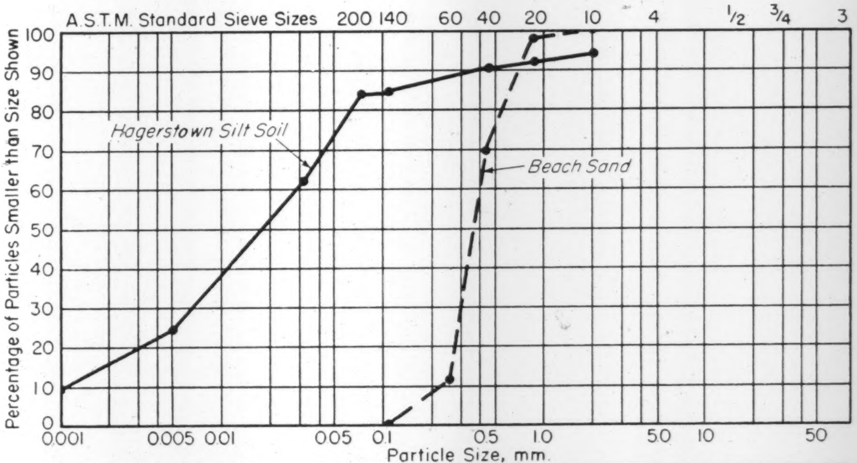
FIG. 1.—Experimental Setup.

Densification:

The soil samples were densified by dropping a standard Proctor (3) hammer of $5\frac{1}{2}$ lb. weight from a height of 12 in. up to 25 times.

Pressure Cells:

Small differential pressure cells (4),



Clay 0-0.005	Silt	Sand	Sand	Gravel	
Colloids 0-0.001	0.005-0.05	Fine 0.05-0.25	Coarse 0.25-2.0	2.0-4.76	4.76-76.2

FIG. 2.—Grain Size Accumulation Curve for Beach Sand and Hagerstown Silt Soil.

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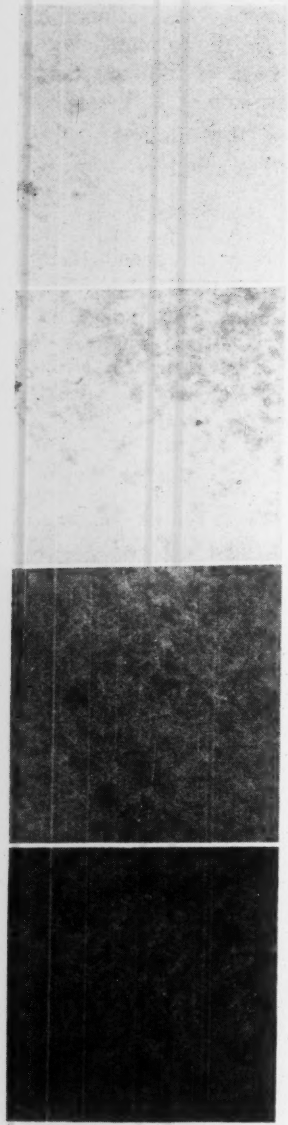
[3] The effect of any remaining scattered radiation has been considered negligible since specimens thinner than 3 in. have not been investigated in these pilot studies. Further experiments are required to check this assumption.

2 in. high and 0.75 in. in diameter, were embedded in the soil sample. A special holder (4) has been developed for the pressure cells to place them automatically and independent of the operator at all times with the same initial pressure on the underlying strata. This holder can be released and withdrawn after the container has been filled with soil to a height considerably above the cell.



	Soil Density, per cent	Photocell Output in 0.1 micro-amp.
(*a*)	66	31
(*b*)	73	28
(*c*)	87	15
(*d*)	94.5	9

FIG. 3.—Radiograph Prints of Hagerstown Silt Soil for Various Densities.

Densitometer:

The radiograph or print was placed on a viewing screen and scanned by a photocell. The output of this cell could be registered by a sensitive galvanometer. The photocell was rigidly fixed above the viewing screen to keep the distance between the light source and the photocell constant at all times. A radiograph or print could be moved between the photocell and viewing screen, following a definite pattern. This pattern (matrix) on the film or print consisted of small dots marked every inch (see Fig. 5) and locating the position of film or print with respect to the fixed photocell. Tests with various uniformly exposed prints have shown that the changes in light transmissibility due to changes in paper thicknesses are negligible.

Exposure and Darkroom Procedure:

Special care was taken that exposure and darkroom procedure was meticulously repeated in every detail for all experiments. As far as X-ray exposure is concerned, voltage, amperage, exposure time, and focal distance were kept constant. In the darkroom, processing of X-ray films took place simultaneously in the same tanks at the same and constant temperature, and for the same length of time. Similarly, all prints were exposed and processed under identical conditions. Positive prints were made of all radiographs to increase the density contrast (6). Instead of using high-contrast-films, high contrast paper prints were made, which do not require viewing screens for interpretation.

The following data, referring to X-ray

exposure and darkroom procedure, were used for almost all experiments:

X-ray exposure.—60 kv., 18 ma., 9 min., at 48 in. focal distance.

Darkroom procedure:

X-ray film.—DuPont 506, no screen; developing time, 7 min. (DuPont X-ray developer); fixing time, 15 min. (Kodak acid fixer with hardener).

X-ray print.—Exposure time, 14 sec.; light source distance, 96 in.; Eastman Kodabromide F-4; developing time, 2 min. (normal Kodak Dektol developer); fixing time, 8 min. (Kodak acid fixer with hardener).

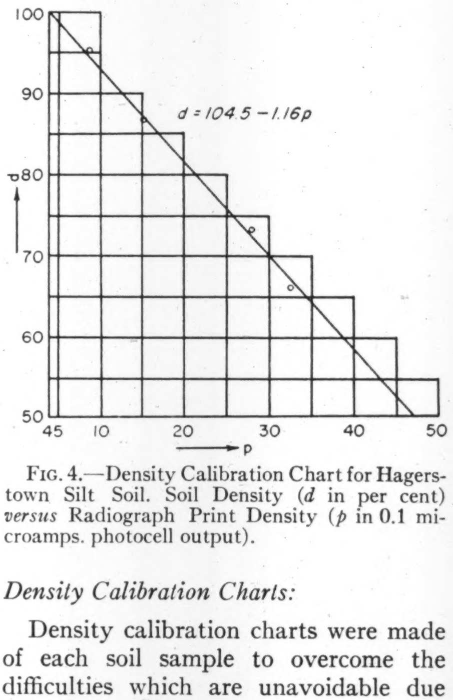


FIG. 4.—Density Calibration Chart for Hagerstown Silt Soil. Soil Density (*d* in per cent) *versus* Radiograph Print Density (*p* in 0.1 microamps. photocell output).

Density Calibration Charts:

Density calibration charts were made of each soil sample to overcome the difficulties which are unavoidable due to variations in chemical composition, atomic structure, grain size, and grain distribution, etc.

TEST RESULTS

Figure 3 shows density calibration images for a Hagerstown silt soil sample with a thickness of 3.11 in. in four stages of density. In Fig. 4, the density of this soil sample, gained from the volumetric change of the soil, is plotted against density of the print (relative transmittance) (Fig. 3), obtained from readings with the densitometer.[4] The soil density *versus* print density curve follows a straight line law of the form:

$$d = 104.5 - 1.16\, p$$

where:

d = Soil Density of the Hagerstown silt soil sample in per cent. (100 per cent soil density represents the maximum compaction within the sample which could be obtained by tamping) and

p = photocell output in 0.1 microamperes.

This relation is purely empirical and requires further investigation. The graph was used as calibration standard for all further Hagerstown silt soil density determinations. Similar graphs are required for any other type soil.

Density Changes Due to Compaction:

In Fig. 5, a positive print of Hagerstown silt soil after compaction is represented. Compaction was produced by dropping the Proctor hammer 15 times from a height of 12 in. at the center of the 3.11 in. thick sample.

The matrixes shown in Fig. 6(*a*) and (*b*) indicate the print density before and after compaction. This print density in photocell output (0.1 microamp) was obtained by scanning the images with the densitometer. All curves represent the loci of equal light transmissibility. Hence, from the density calibration chart for Hagerstown silt soil (Fig. 4) the isopycs, coined from the Greek word

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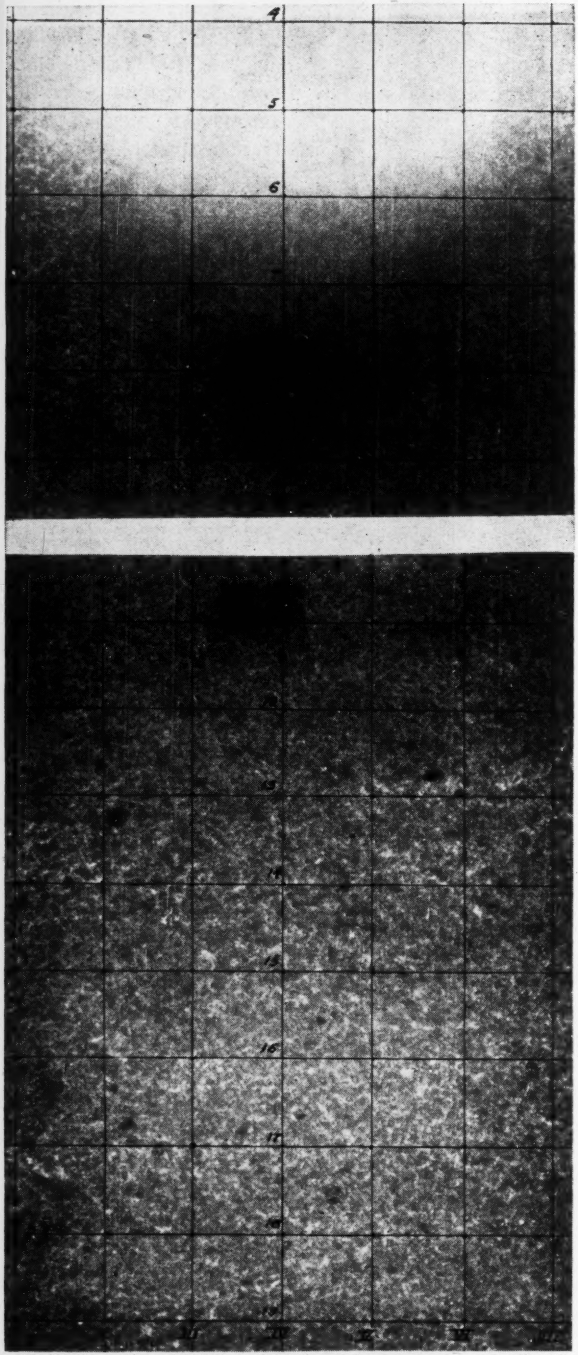
[4] The maximum density obtained by tamping was designated as 100 per cent and the corresponding change in volume (settlement) recorded. Less compaction was produced by reducing the amount of tamping and the obtained volume changes used to calculate intermediate density values in per cent.


4
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III
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V
VI
VII

4 to 19: Abscissa of Matrix (distance in inches below not compacted soil surface).
I to VII: Ordinates of Matrix (distance in inches from left side of soil sample).

FIG. 5.—Radiograph Print of Hagerstown Silt Soil After Compaction.

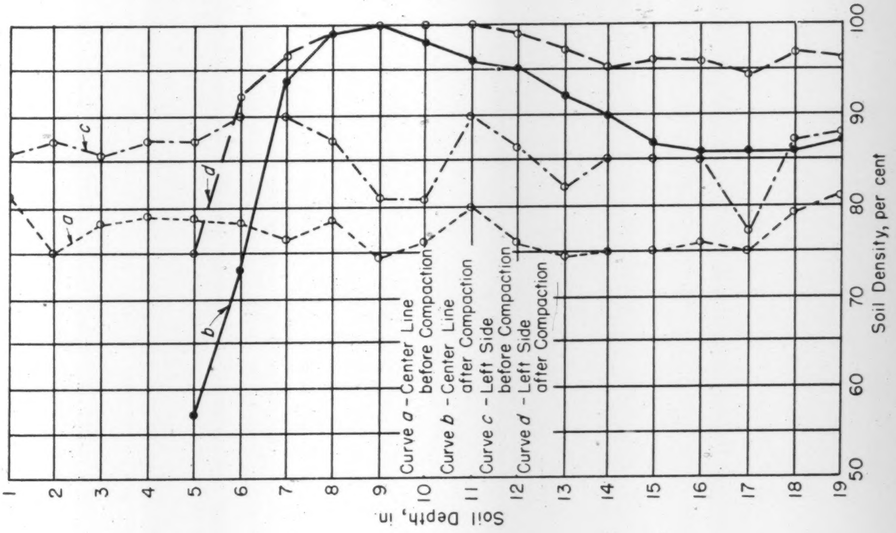


FIG. 7.—Soil Density Distribution for Hagerstown Silt Soil Along Center and Left Side of Container Before and After Compaction.

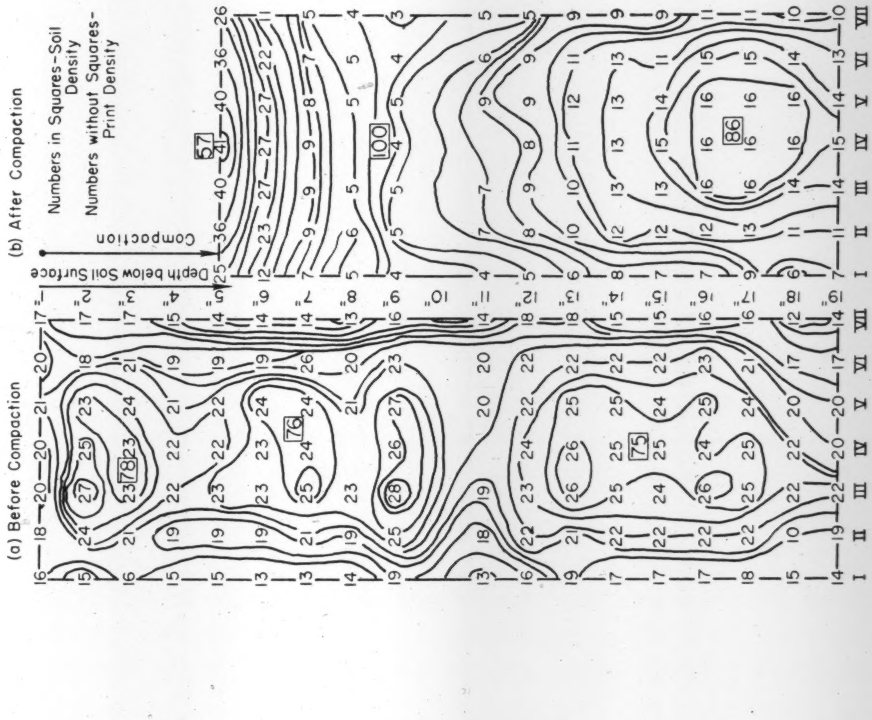
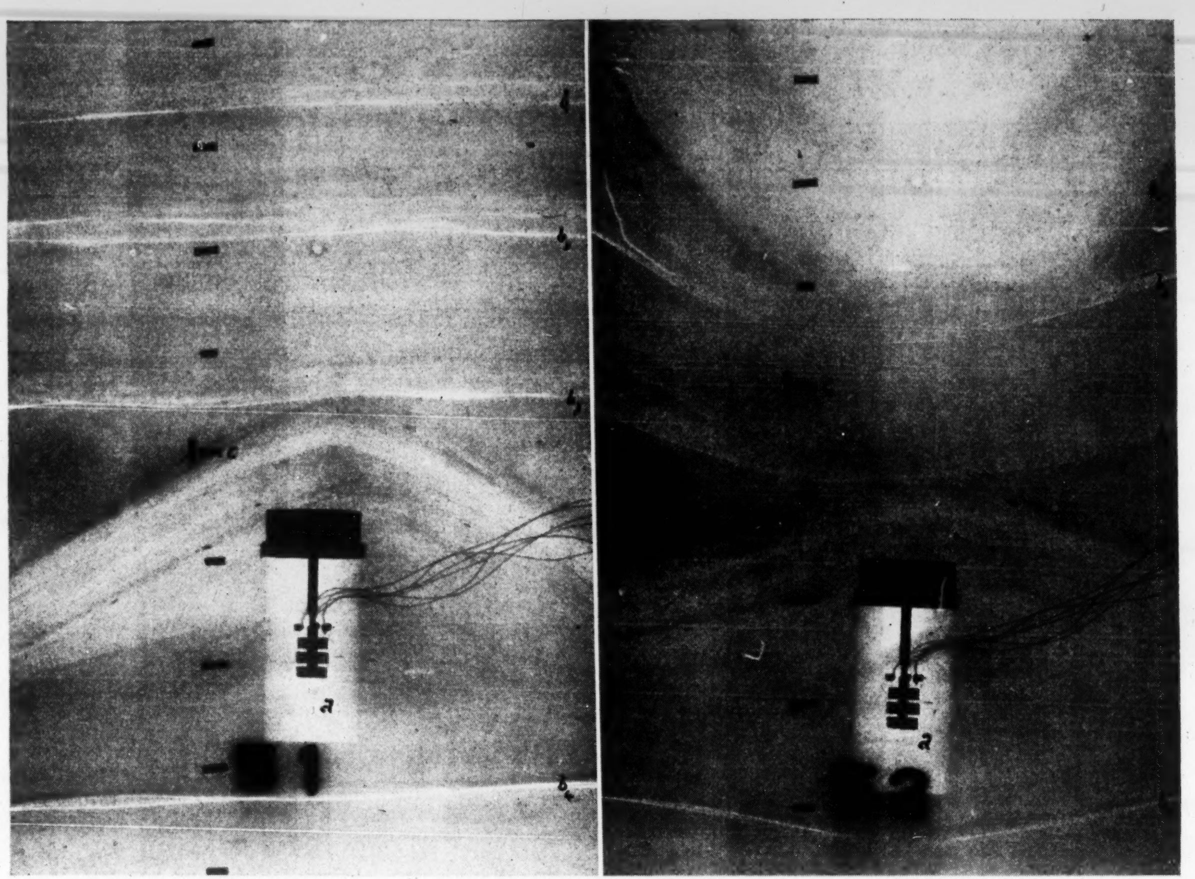


FIG. 6.—Density Matrix and Isopycs for Hagerstown Silt Soil Before and After Compaction.



Before Compaction.

After Compaction.

FIG. 8.—Radiograph Print of Beach Sand with Imbedded Differential Pressure Cell (*a*), Aluminum foils (*b*), and Lead markers every inch (*c*).

"pyknos," that is the loci of constant soil density, could be derived. These isopycs are identical in shape with the loci of equal light transmissibility. Thus the soil density at any point in the sample can be determined.

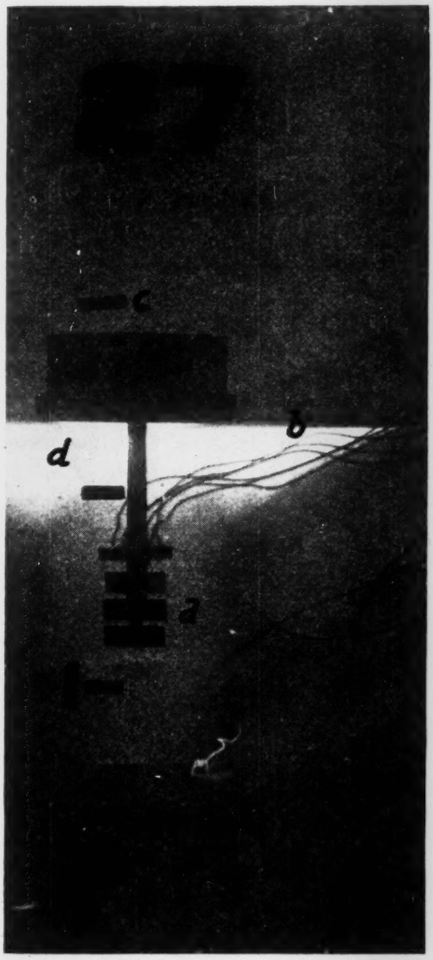
The sensitivity of this radiographic method can be visualized by comparing the change of a medium density area near the bottom of the container before and after compaction (Fig. 6(*a*) and (*b*)). An hourglass-shaped area (major axis vertical) of isopyc 75 Fig. 6(*a*) degenerates into a circular shape that is, (isopyc 86, Fig. 6(*b*)), while the geometrical center of this area moves approximately 2 in. downward.

All experimental values could be reproduced with an accuracy of ±2 per cent.

Further results of this determination are plotted in Fig. 7, showing the soil density distribution before and after compaction along ordinates I and IV, that is, near the left wall and in the center of the container. From these curves, the effect of the wall friction becomes obvious. The maximum compaction density (100 per cent) around 10 in. below the surface of the soil sample as produced by tamping the surface seems not to be reduced by confining effects.

Motion of Pressure Cell Due to Compaction:

Figure 8 is a positive print of radiographs of beach sand samples with a thickness of 3.11 in. before and after compaction. Compaction was produced in the same manner as described previously. Embedded in the soil is a differential pressure cell and four aluminum foils. The deformation of the aluminum foils and the sinking of the pressure cell for more than one inch after compaction is clearly visible. The upper foil broke during compaction due to excessive strain. The streaks of light bending around the pressure cell are caused by irregularities while pouring the sand into the container.



(*a*) Pressure cell.
(*b*) Fin Attached to (*a*).
(*c*) Lead markers every inch.
(*d*) Void.

Fig. 9.—Radiograph Print of Beach Sand and Embedded Differential Pressure Cell with Attached Spring Steel Fin.

Size Effect of Pressure Cell:

Figure 9 is a positive print of a radiograph of a beach sand sample of 3.11 in. thickness before compaction showing the effect of a horizontal fin of 3 in. diameter attached to the pressure cell. The bright area under the fin indicates the formation of a void produced by the extension of the pressure cell in the horizontal direction. Discontinuities of similar type might easily distort the output of pressure cells. No voids could be observed under the bottom of pressure cells having a diameter of 0.75 in. only (see Fig. 8).

ANALYSIS

The following conclusions might be drawn from these experiments, keeping in mind that the unavoidable friction of the container walls will distort the findings to a certain extent.

Exposure and Darkroom Technique:

In order to produce the maximum contrast in the radiograph due to changes in density of the soil sample, the following technique seems recommendable:

Soft X-rays, that is voltages not higher than 100 kv., yield satisfactory contrasts. A distance between focal spot and X-ray film of more than 4 ft. will prevent undesirable contrast changes due to increased length of the X-ray path at the fringes of the image. The two pictures, before and after compaction, have to be taken and processed under absolutely identical conditions. Successive exposures by covering first one side of the film with a lead mask before compaction and then masking the other half of the film after compaction are preferable. Developing and fixing the exposed films or prints simultaneously is an important prerequisite.

Densitometer Readings:

The distance between photocell and light source has to be kept constant at all times. Leaving the photocell at a fixed point and moving the matrix marking the scanning points on the image will facilitate this requirement. The photographic density contrast can be substantially increased by making positive contact prints. X-ray film and print paper are used advantageously in the steepest straight-line range of their characteristic (log relative exposure density) curves (6).

Density Changes:

Special care has to be taken while pouring the soil into the container (see Fig. 8). In particular for cohesive soil, it is difficult to obtain a uniform density distribution even before compaction (see Figs. 6(*a*) and 7(*a*) and (*c*)). An evaluation of the isopycs near the container walls will permit, to a certain extent, the isolation of the effect due to nonavoidable wall friction (see Figs. 6 and 7). The known phenomenon, that impact loads cause the maximum densification at some distance below the soil surface and a pronounced dedensification close to the surface, could be reproduced (see Fig. 7(*b*) and (*d*)).

Differential Pressure Cells:

Two rather obvious facts have been demonstrated: first, that small bodies, such as pressure cells, embedded in the soil participate to a substantial degree in the motion of the surrounding soil particles (see Fig. 8), and, second, that the smaller the cell, the less the continuity of the soil will be interrupted. It seems that the positioning, or the initial pressure with which the cell is placed on the underlying strata, is of particular importance. This holds true not only for the prevention of tilting during compaction, but also for the resultant output of the pressure cell.

Future Developments:

An interesting development of X-ray tubes, so-called Oxtail Tubes (2), with long tubular anode extensions might lead to a practical application of the X-ray method in the field. Another possibility might be the application of radioactive-isotopes, for example radioactive cobalt (Co 60) with a gamma ray emanation per unit weight several times that of radium (7, 8)[5] (see Fig. 10). It must be well understood, however, that such a method can be justified only where undisturbed soil samples cannot be obtained otherwise. This might be of particular importance for dry noncohesive soils (sand).

SUMMARY

The developed X-ray technique to measure quantitatively and qualitatively changes in density of soil samples from densitometer readings seems to yield satisfactory results.

The effect of compaction with respect to shape and motion of pressure cells buried in soil samples could be determined.

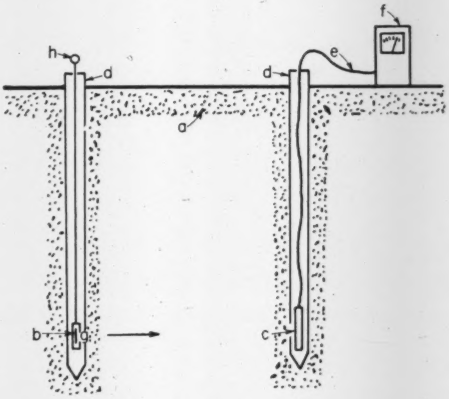
Further theoretical and experimental investigations are required with respect to the described technique as well as to correlate differential pressure cell output with certain effects due to soil compaction produced by static and dynamic loads.

X-ray tubes with long tubular anode extensions or radioactive-isotopes might lead to possible future applications of the X-ray method in the field where undisturbed soil samples cannot be obtained otherwise.

Acknowledgment:

The investigation is part of a Highway research project on "Dynamic Properties and Consolidation of Soils," sponsored jointly by the New Jersey State Highway Department, the Public Roads Administration, and the State University of New Jersey (Rutgers University).

All tests were made at the Engineering Experiment Station, College of Engineering, the State University of New Jersey. The constructive criticism of J. Slade, Director of the Engineering Experiment Station, is thankfully acknowledged.



(*a*) Soil.
(*b*) Radioactive isotope in lead shielded capsule.
(*c*) Electrometer tube and ionization chamber.
(*d*) Steel pipe.
(*e*) Shielded cable.
(*f*) Radiation indicator.
(*g*) Ray emission window (normally closed).
(*h*) Opener for ray window (remote control).

FIG. 10.—Soil Density Determination with Radioactive Isotopes.

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[5] Pilot experiments with Co 60 have yielded rather encouraging results. Reductions of 50 per cent in transmission of gamma rays were measured before and after compaction of Hagerstown silt soil samples varying in thickness from 6 in. to 50 in.

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DISCUSSION

Mr. Gregory P. Tschebotarioff[1] (*presented in written form*).—The paper treats a subject of considerable theoretical and practical interest and the authors are to be complimented on their efforts to solve the problem. A thorough job has been done within the limits imposed by the reduced scale of the experiments. However, no numerical data are presented in the paper which would permit one to judge whether optimism is really justified concerning the possible application of the method in the field at full scale. The nature of the difficulties encountered by previous investigations should be mentioned in this connection.

X-rays have been used in the past for a somewhat similar problem, namely, to study the deformations of a sand mass under load. Such tests were performed by Emil Gerber at Zurich in 1929.[2] Small (3-mm. diameter) lead spheres were embedded in the sand at several elevations under the center line of a 3-in. steel plate which rested on the surface. The width of the box, which was filled with sand, was 20 cm. (8 in.). A photograph was taken *before* a load was applied at the sand surface and the exposure was repeated on the same photographic plate *after* application of the load. The 8-in. thickness proved to be the maximum which the X-rays of the apparatus available at the time could penetrate effectively. However, even so the photographs were insufficiently clear for reproduction, and the accompanying diagram, Fig. 11 had to be reconstructed from them. The lead spheres immediately under the steel surface plate did not show up at all on the second photograph because of interference induced by that plate.

I am not aware that any other X-ray experiments for a sand thickness greater than 8 in. have ever been successfully performed. It would therefore be interesting to know on what the authors base their hopes that the success of their experiments with a much smaller thickness of sand,—3 in. only—can be duplicated in the field where many times larger thicknesses will be involved. After all not only the intensity of the rays decreases with the square of the distance from the source but also the absorption by the soil follows an exponential relationship.

The proposal to use radioactive cobalt instead of X-ray tubes appears to be a step in the right direction. Mr. Milton G. White, Professor of Physics at Princeton University, whose opinion I have sought on this entire problem, suggested that instead of lowering a photographic plate into one of the bore holes a specially designed counter of gamma rays might possibly be employed and might prove to be more sensitive and practical in operation. Nevertheless, the distance between the bore holes could even then hardly be greater than some 30 to 40 in. if differentiation between various states of density of a sand is to be possible.

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[1] Associate Professor of Civil Engineering, Princeton University, Princeton, N. J.

[2] "Untersuchugen uber die Druckverteilug im ortlich belasteten Sand," by Emil Gerber Doctorate dissertation, Zurich, Switzerland, 1929.

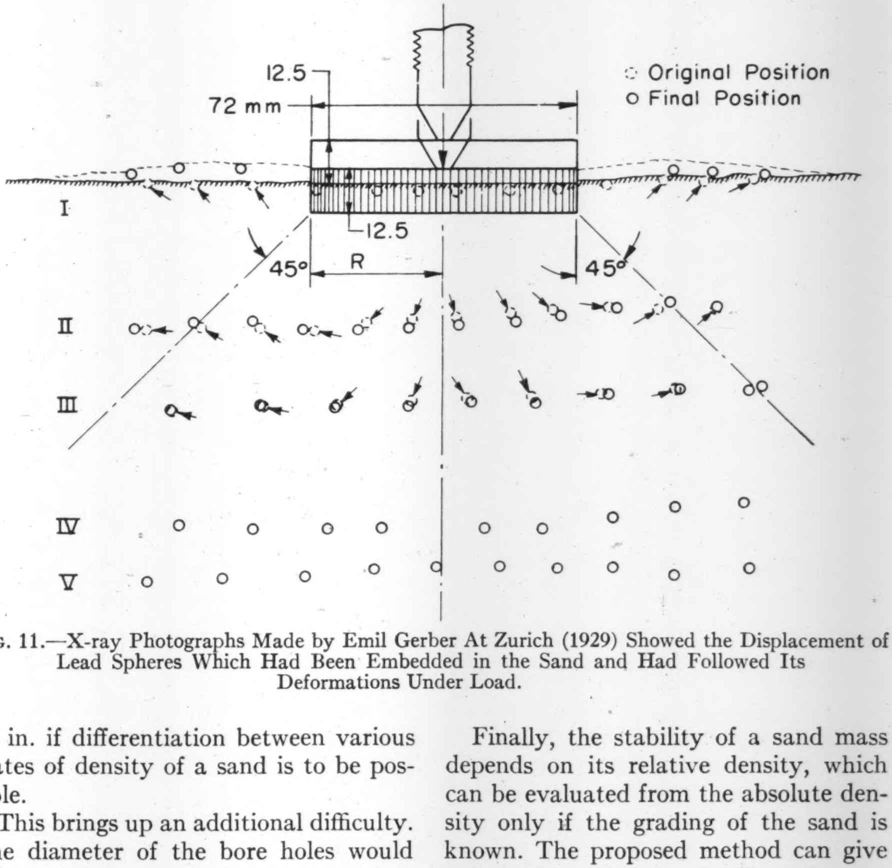

FIG. 11.—X-ray Photographs Made by Emil Gerber At Zurich (1929) Showed the Displacement of Lead Spheres Which Had Been Embedded in the Sand and Had Followed Its Deformations Under Load.

This brings up an additional difficulty. The diameter of the bore holes would have to be at least 4 in. In dry sand they would have to be lined with a casing. The sinking of two 4-in. casings at some 30 in. distance from each other would be liable to change the original density of a loose sand between them at a depth exceeding some 5 or 6 ft., if not even at shallower depths.

A second difficulty appears to have been realized by the authors who emphasize that their method is best suited for dry sands. This is definitely the case since presence of moisture will have the same effect as an increase in the density of a dry sand—water in the voids will offer greater resistance to the passage of rays than will air in the voids.

Finally, the stability of a sand mass depends on its relative density, which can be evaluated from the absolute density only if the grading of the sand is known. The proposed method can give values of the absolute density only.

Summing up, it would appear that in the field the proposed method could give practically usable results only with sand of known grading and moisture content and only at very shallow depths.

MESSRS. D. BERDAN AND R. K. BERNHARD (*authors' closure*).—The question has been raised concerning the field application of X or gamma rays to determine soil densities. Some anticipated difficulties are mentioned, in particular the possible distance of detectable ray-transmission through the soil was questioned.

As pointed out on page 1329 the container had an inside cross-section of 9 by 9 in. No difficulties arose in making radiographs up to the full capacity of the container. Samples with 3.11 in. thickness were used for some of the tests to reduce the exposure time.

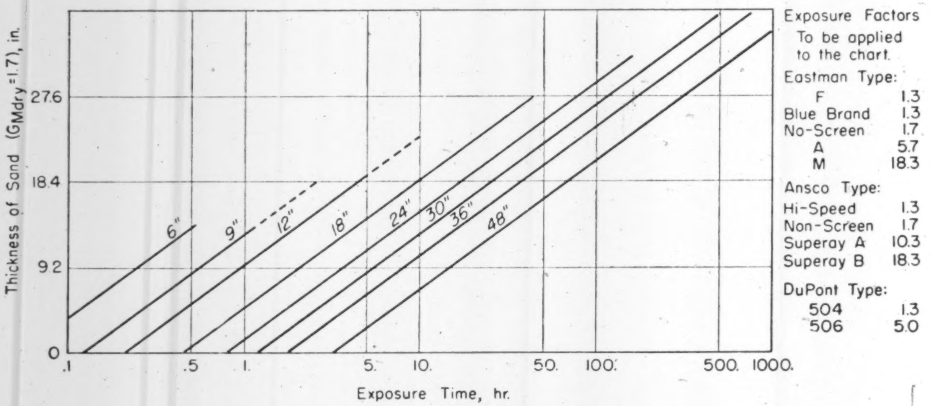

FIG. 12.—Determination of Exposure Times.

Source: 100 mg. Radium. Film: Eastman Industrial Type K. Distance: Radium to Film, as Indicated. Film Density: 1.0 H. and D. Intensifying Screens: Lead-Alloy Foil. Development: Twice Normal Development Time.

The experiments by E. Gerber (1929), as mentioned by the discusser, are superceded by new tests made by O'Dea (1949)[3] and Belcher and associates (1950).[4] Using a gamma-ray emanating radium source, O'Dea succeeded in making radiographs through soil samples of approximately 20-in. thickness. He developed an exposure time chart for dry soil samples up to 27.6 in. thickness as shown in the accompanying Fig. 12.

Belcher and associates determined soil densities, including the relation of moisture content, by means of a radium source in the laboratory and in the field. Figure 13 represents a soil density *versus* counts per minute chart for soils with various moisture contents.

Furthermore they experimented with polonium-beryllium as a neutron source to determine moisture contents.

In both cases, back scattering methods were used, with the advantage that transmitter and receiver could be inserted in one and the same tube. The maximum outside diameter of this tube was 1.25 in., the greatest depth below the surface 9 ft. Significantly larger depths will require an adjustment of the circuit.

Belcher and associates summarize some of their findings as follows:

> "The gamma ray scattering method proves to be a reliable method for determining density without disturbing the soil mass, at least if care is taken that the placing of the tube does not disturb the soil mass near the tube. The precision, at present, is ±2 per cent per cu. ft.
>
> "The neutron scattering method proves to be a reliable method for determining, with-

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[3] J. J. O'Dea, Jr., "An Experimental Investigation of the Vibratory Characteristics of Sand," thesis presented to Faculty of Renssalaer Polytechnic Institute, Troy, N. Y., May, 1949.

[4] T. R. Guyhendall, H. S. Sack, D. J. Belcher, "The Measurement of Soil Moisture and Density," College of Engineering, Cornell University, Ithaca, N. Y., Report of Civil Aeronautics Authority, April, 1950.

out disturbing the soil mass, the moisture content. The precision is ±1 lb. water per cu. ft. or better, corresponding to about ±1 per cent moisture content.

"It can be said that both methods have proven themselves in laboratory and field tests and promise to become satisfactory and very useful tools for the measurement and continuous recording of density and moisture content of soils."

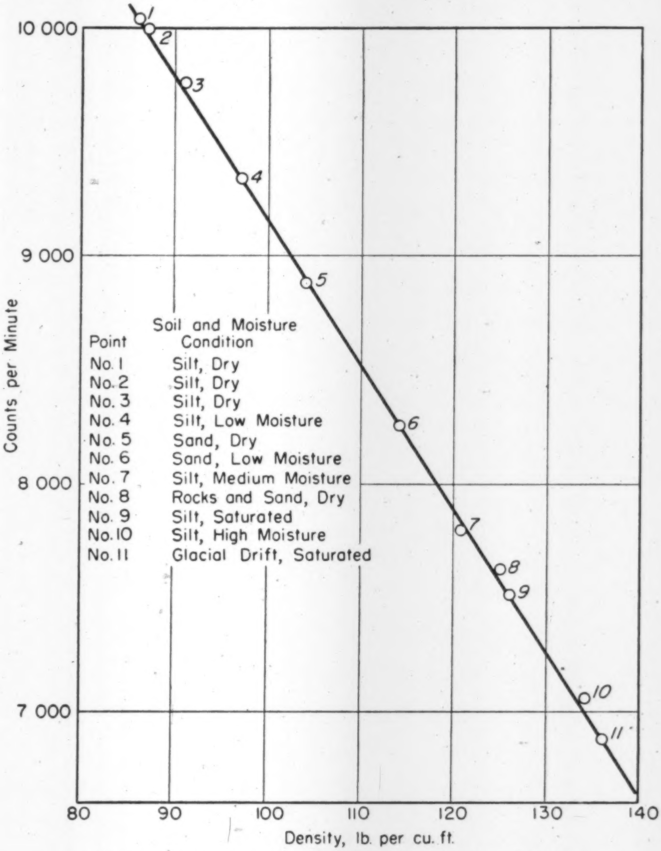

FIG. 13.—Counting Rate *versus* Wet Density of Soils.

With respect to the proposed direct transmission method by means of radioactive cobalt, the dicusser has misinterpreted Fig. 10. The authors show a gamma ray pick-up unit in the lower end of the right-hand tube, connected to an indicating instrument above the ground, and not a photographic plate. As reported in the footnote on page 1337, pilot experiments with Co 60 showed a reduction of 50 per cent in gamma ray transmission before and after compaction of Hagerstown Silt Soil varying in thickness from 6 in. to 50 in. It has to be kept in mind that only a rather weak radioactive source (18.5 millicurie of Co 60) was available, and furthermore that the pick-up unit had a low sensitivity (50 millirep per hour). Nevertheless the assumption seems justified that substantially larger distances than 50 in. can be penetrated if so desired.

The authors agree with Mr. Tschebotarioff that the grain size distribution of of the soil cannnot be determined by either method which basically integrate the soil volume under investigation. The primary objective of these X- and gamma-ray studies was the determination of changes in absolute density, with the secondary aim to disturb the soil the least possible amount.

No methods seem to be available at present which might solve this problem better or even similarly effective.

The authors appreciate very much indeed Mr. Tschebotarioff's discussion. They also are sincerely thankful for having obtained the permission from Messrs. O'Dea, Kilcawley, Belcher and associates to report some of their highly significant results.

MR. D. T. O'CONNOR[5] (*by letter*).—The authors of this paper are to be congratulated for their ingenious approach to this soil mechanics problem. Too often we associate the use of X-rays with production inspection problems, particularly casting and weld inspection. Yet the unique properties of X-rays should suggest their use in scores of engineering and development problems. X-rays properly used can be a powerful tool for the collection of primary engineering data as these authors have so amply demonstated.

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[5] Chief, Radiological Section, U. S. Naval Ordnance Laboratory, White Oak, Silver Spring, Md.

THE EFFECT OF ROCK CONTENT AND PLACEMENT DENSITY ON CONSOLIDATION AND RELATED PORE PRESSURE IN EMBANKMENT CONSTRUCTION*

By Harold J. Gibbs[1]

Synopsis

The placement conditions of embankment materials for high earth dams have been found to require careful control so as to prevent excessive pore pressure and provide a good quality of stability. Greatest emphasis has been placed on the finer soils. The concept which has been developed for these soils implies that impervious materials placed at relatively high moisture contents and which will have large consolidations will develop excessive pore pressures when loaded and will cause a greatly reduced stability of the structure. If this concept is carried further into the range of materials containing considerable amounts of coarser, rock-size particles, there has been reason to believe that similar problems would arise. A clearer understanding of such a problem was believed to be necessary. This paper describes the laboratory tests for such an analysis on embankment material containing coarse size particles up to 3 in. in size. The results of these tests demonstrate the effect that changes in rock content have on consolidation and the subsequent effect on possible pore pressure development.

The problem of consolidation and stability for materials containing considerable amounts of rock arose in connection with the construction of Jackson Gulch Dam, a high rolled-earth embankment which the Bureau of Reclamation is constructing at Mancos, Colo. The dam consists of an impervious center zone (Zone 1) of fine material and an outer "Zone 2" material, which contained a considerable amount of rock up to 3 in. in size. This "Zone 2" material was a well-graded gravel with a considerable amount of excellent clay binder. The laboratory had previously studied the "Zone 1" material for placement control and had established limits for desirable placement moisture to avoid excessive pore pressure development in this soil. However, it was observed during construction that the "Zone 2" material also had placement characteristics which could lead to excessive pore presure development and a subsequent decrease in stability.

The compacted densities of the soil-rock embankment obtained during construction were lower than those anticipated, because of the high rock contents which were encountered. Furthermore, the compacted material had the appearance of being quite impervious since the fine portion was an excellent clay binder. Because of the low degree of compaction, consolidation of considerable amounts was anticipated; and, because of the low-permeability appearance of the compacted mass, a very slow draining condition was expected to exist during the consolidation period. Therefore, it was felt that excessive pressure development in the pore fluid, when the material was loaded, might become an important problem in this structure. A detailed consolidation study was made on this material containing rock up to 3 in. in size to determine the pore pressure characteristics in relation to degrees of compaction, rock contents, loading pressures, and moisture contents which were similar to those being obtained in the field during the construction of the dam.

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The stability of high rolled-earth dams, which are constructed of materials having relatively high moisture contents and having high consolidation properties, has been found to be greatly reduced by the development of pore-fluid pressure. Pore pressure (also termed excess hydrostatic pressure) develops in the mass as it consolidates from loading during construction, and a corresponding reduction in effective particle contact pressure is produced. Construction pore pressures have previously been estimated on the basis of rational formulas and laboratory tests[2, 3] and field observations.[4] A means of establishing placement control limits for fine material by laboratory tests to limit pore pressure has been developed.[5] The magnitude of the pore pressure is dependent upon the compressibility of the pore fluid (air and water), the volume change of the mass under load, and the permeability of the mass and surrounding material. Therefore, the effect of pore pressure on stability assumes increased importance as the fill loads and consolidation become large, as the permeability becomes small, and when placement moisture contents are relatively high.[5]

LABORATORY TESTS

The detailed laboratory investigations reported herein were made on a 1000-lb. sample of pit run, Zone 2 material of 3-in. maximum size. Since the degree of compaction, the consolidation, and the imperviousness of the material were items causing greatest concern with relation to pore pressure development, a testing program consisting of many one-dimensional consolidation and permeability tests was conducted on the soil containing various amounts of rock compacted to densities of 80, 90, and 100 per cent of the theoretical field density. The rock contents varied from 25 to 70 per cent, and loading pressures of 50, 100, and 150 psi. were used.

For control purposes and basic data, the sample was visually classified and standard laboratory tests were made to determine the gradation and specific gravity of the total material, and the moisture-density (compaction) characteristics of the minus No. 4 fraction of the total material. The moisture-density test used was similar to the A.S.T.M. Tentative Method D 698[6] except that a $\frac{1}{20}$ cu. ft. compaction cylinder was used and the height of hammer drop was 18 in. In order to simulate the compaction of the fine material secured by the heavy rollers on this project, 35 blows per layer were used.

Consolidation Equipment and Testing Procedure:

The consolidation testing equipment involved two sizes of apparatus: an 8-in.

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[2] L. W. Hamilton, "The Effects of Internal Hydrostatic Pressure on the Shearing Strength of Soils," *Proceedings*, Am. Soc. Testing Mats., Vol. 39, p. 1100 (1939).

[3] J. W. Hilf, "Estimating Construction Pore Pressure in Rolled Earth Dam," *Proceedings*, Second International Conference on Soil Mechanics, Paper IV b 5, Vol. III (1948).

[4] F. C. Walker and W. W. Daehn, "Ten Years of Pore Pressure Measurements," *Proceedings*, Second International Conference on Soil Mechanics, Paper IV b 7, Vol. III (1948).

[5] W. G. Holtz, "The Determination of Limits for Control of Placement Moisture in High Rolled-Earth Dams," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 1240 (1948).

[6] Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 42 T), 1949 Book of A.S.T.M. Standards, Part 3, p. 1180.

diameter cylinder containing a test specimen 3 in. high for testing 1-in. maximum size material and a 20-in. diameter cylinder containing a test specimen 8 in. high for testing 3-in. maximum size material. These cylinders were adapted from the standard permeability-settlement testing equipment for embankment materials which are presently used in the Bureau of Reclamation laboratory. Figure 1 shows the details of the 8-in. diameter apparatus which has a hydraulic jack for applying the load and a spring that is clamped in position for retaining the load. Figure 2 shows the 20-in. diameter apparatus being loaded by a 120,000-lb. testing machine, and this apparatus has four springs for retaining the load. Both pieces of apparatus are of similar construction.

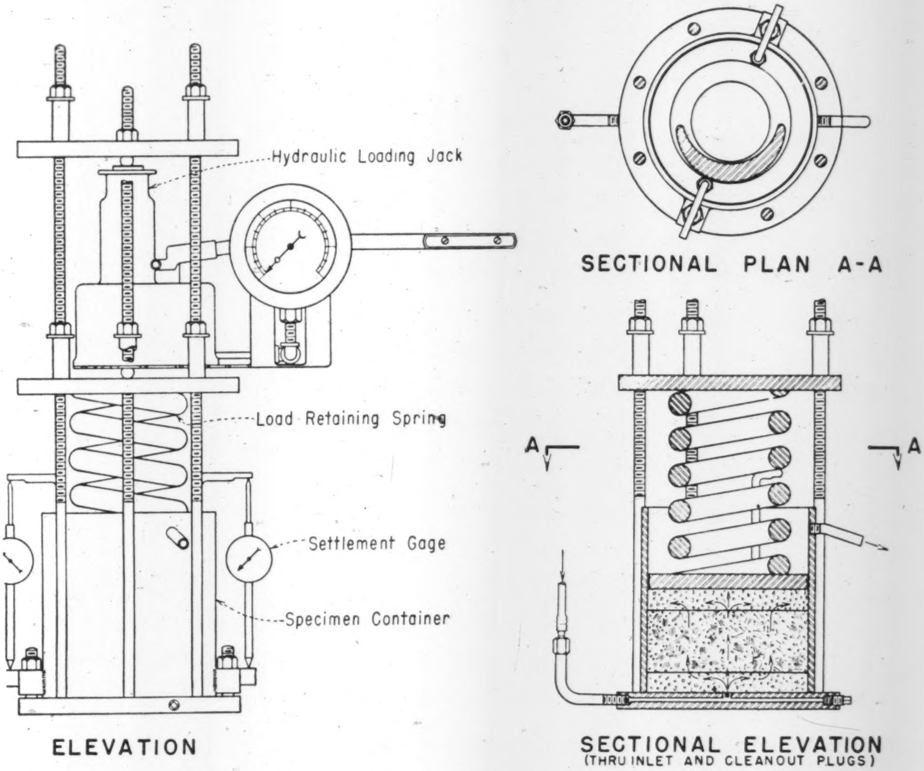
Hydraulic Loading Jack
Load Retaining Spring
Settlement Gage
Specimen Container
ELEVATION
SECTIONAL PLAN A-A
A
A
SECTIONAL ELEVATION
(THRU INLET AND CLEANOUT PLUGS)

FIG. 1.—8-in. Diameter Consolidation Apparatus.

The method of test involves a consolidation determination for test specimens which are restrained laterally and loaded and drained axially. Top and bottom porous mediums (porous plates in the case of the 8-in. cylinders and porous plates plus a $\frac{1}{2}$-in. layer of coarse sand in the case of the 20-in. cylinders) allow complete drainage during consolidation. Strain gages mounted on the sides of the cylinders indicate the movement of the top bearing places and measure the thickness change of the specimens during load. Permeameter tubes connected with the bottom porous plate, are used to saturate the specimens and obtain an indication of the permeability characteristics. Remolded specimens of varying rock content were compacted into these cylinders at desired densities and moisture contents. The specimens were placed in two layers which were thick enough to allow compaction of the maximum size rock.

The testing program required many consolidation tests. Because of the limitations in time and equipment available for testing, it was not possible to use the large apparatus with 3-in. maximum rock size for the entire testing program. A modified plan was adopted which made use of a great number of tests with smaller 8-in. diameter equipment with material containing 1-in. maximum size rock. Sufficient tests using the larger equipment and 3-in. maximum size rock were made for correlation purposes.

For this modified plan, three grain size distributions were selected. These are shown in Figs. 3, 4, and 5. The term "gradation series" as used in this paper refers to the series of tests made on a material of a particular grain size distribution. For tests in the large cylinder, material of 3-in. maximum size was used; for tests in the smaller cylinders, material with the same grain size distribution was used, but the rock from 1 to 3 in. in size was excluded. Each of Figs. 3, 4, and 5 show a particular grain size distribution for maximum sizes as used in the tests.

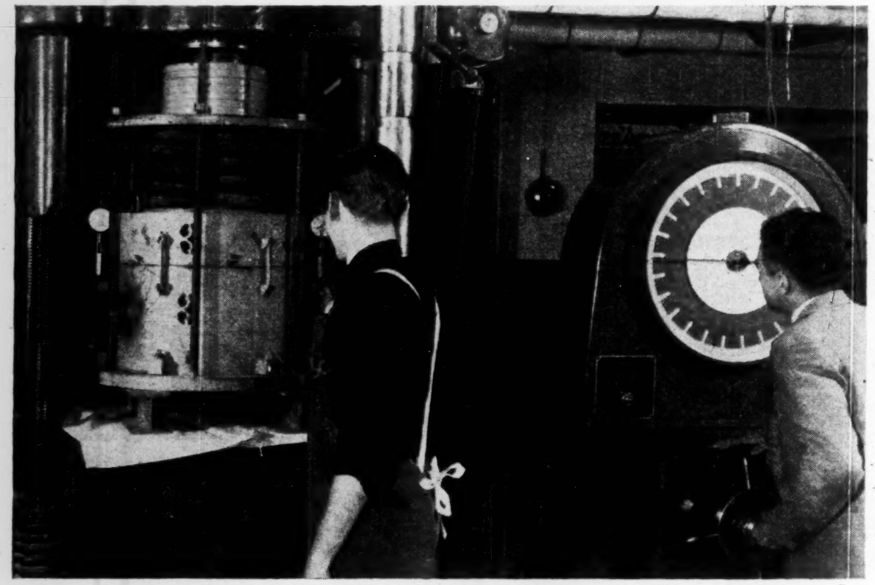

FIG. 2.—20-in. Diameter Consolidation Apparatus.

DISCUSSION OF TEST RESULTS

A schedule of the consolidation tests is shown in Fig. 6. This schedule includes three series of tests, each for a different grain size distribution so that a variation in rock content is obtained. Each series includes consolidation tests for three degrees of placement compaction. Each placement condition was studied for three loadings for the 8-in. cylinder tests and one loading, 100 psi., for the 20-in. cylinder tests.

Moisture-Density Tests on the Fine Material:

The results of the moisture-density tests on the fine material are shown in Fig. 7. The 35-blow per layer compaction was used, as it represented the energy of roller compaction which was being used in the field. The maximum density and optimum moisture of this material were 104.7 lb. per cu. ft. and 20.0 per cent, respectively. A placement condition of 2 per cent less than optimum, which was in the approximate range of desirable field control, was selected for the consolidation studies. This gave a placement moisture of 18.0 per cent and a corresponding placement density of 103.5 lb. per cu. ft.

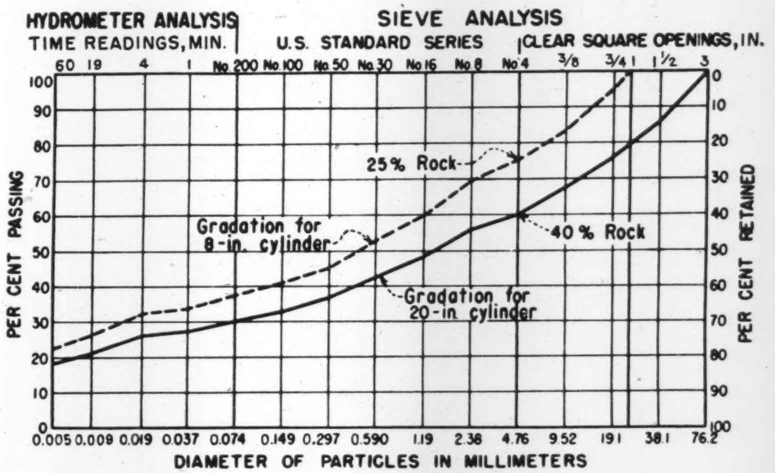

FIG. 3.—Gradation for Series 1.

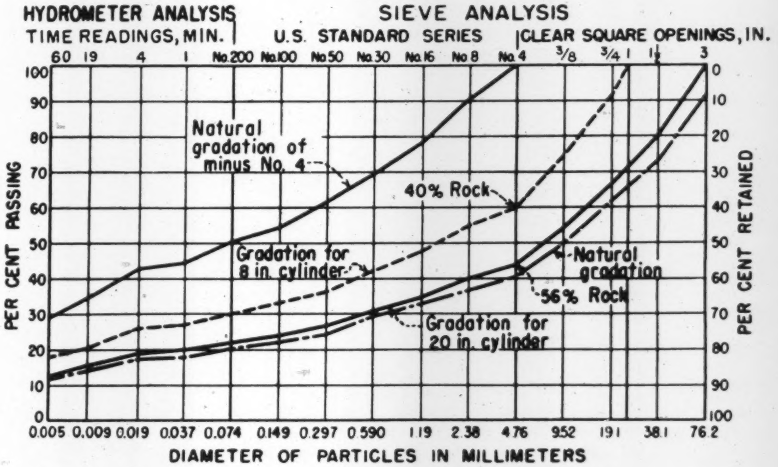

FIG. 4.—Gradation for Series 2 Natural Rock Mixture.

Theoretical Placement Densities for the Material Containing Rock:

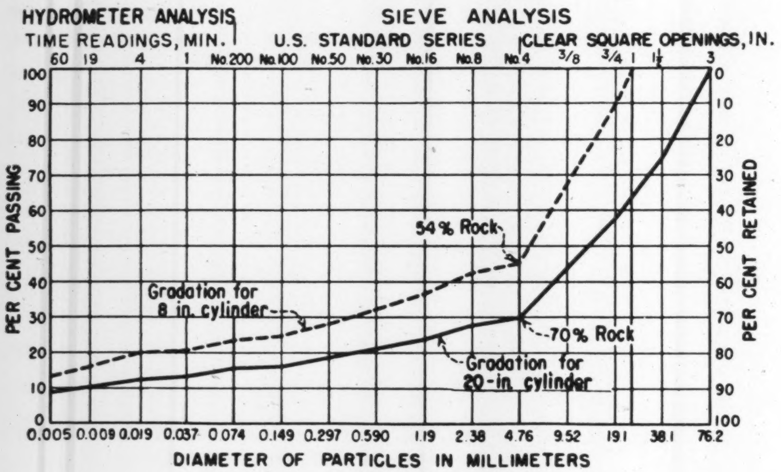

FIG. 5.—Gradation for Series 3.

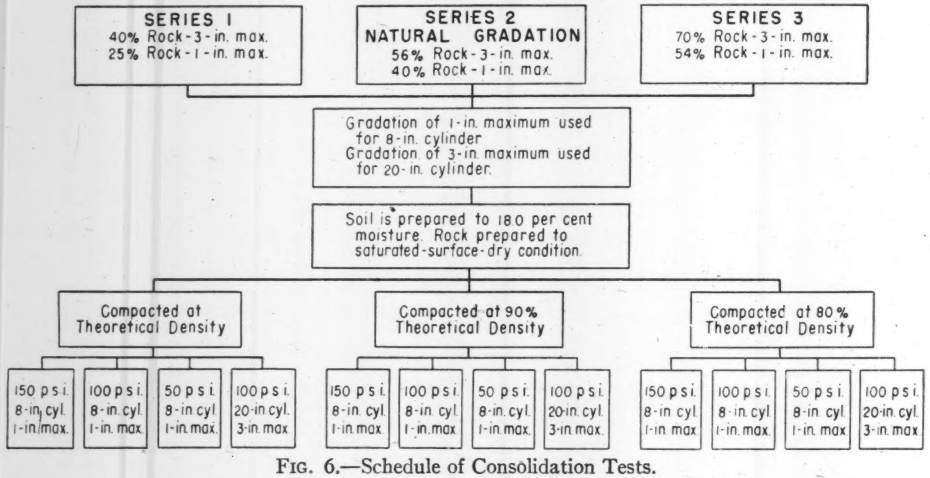
SERIES 1
40% Rock - 3 - in. max.
25% Rock - 1 - in. max.
SERIES 2
NATURAL GRADATION
56% Rock - 3 - in. max.
40% Rock - 1 - in. max.
SERIES 3
70% Rock - 3 - in. max.
54% Rock - 1 - in. max.
Gradation of 1-in. maximum used for 8-in. cylinder
Gradation of 3-in. maximum used for 20-in. cylinder.
Soil is prepared to 18.0 per cent moisture. Rock prepared to saturated-surface-dry condition.
Compacted at Theoretical Density
Compacted at 90% Theoretical Density
Compacted at 80% Theoretical Density
150 p.s.i. 8-in. cyl. 1-in. max.
100 p.s.i. 8-in. cyl. 1-in. max.
50 p.s.i. 8-in. cyl. 1-in. max.
100 p.s.i. 20-in. cyl. 3-in. max.
150 p.s.i. 8-in. cyl. 1-in. max.
100 p.s.i. 8-in. cyl. 1-in. max.
50 p.s.i. 8-in. cyl. 1-in. max.
100 p.s.i. 20-in. cyl. 3-in. max.
150 p.s.i. 8-in. cyl. 1-in. max.
100 p.s.i. 8-in. cyl. 1-in. max.
50 p.s.i. 8-in. cyl. 1-in. max.
100 p.s.i. 20-in. cyl. 3-in. max.

FIG. 6.—Schedule of Consolidation Tests.

Figure 8 is a graph showing the theoretical densities for various percentages of rock and degrees of placement compaction. The maximum soil rock density is based on a density of 103.5 lb. per cu. ft. for the fine material and a specific gravity of 2.52[7] for the rock. This is referred to as the 100 per cent theoretical density in this paper for the rock contents under consideration. The lower degrees of compaction are shown as 90 and 80 per cent of this density. That is, the theoretical 100 per cent density is the unit weight of the soil-rock mixture when the minus No. 4 soil completely fills the space between the rock particles and soil has a density of 103.5 lb. per cu. ft.

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[7] The specific gravity of 2.52 which was used for the rock material in this study is the "bulk specific gravity (saturated surface-dry basis)" as described in A.S.T.M. Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 42), 1949 Book of A.S.T.M. Standards, Part 3, p. 782. This would be the specific gravity of the rock material when the normal voids contained water. Later opinions regarding this item have indicated that a "bulk specific gravity" as described in the above test would have been more proper as it would be the specific gravity of the dry rock and would indicate a slightly lower theoretical density curve.

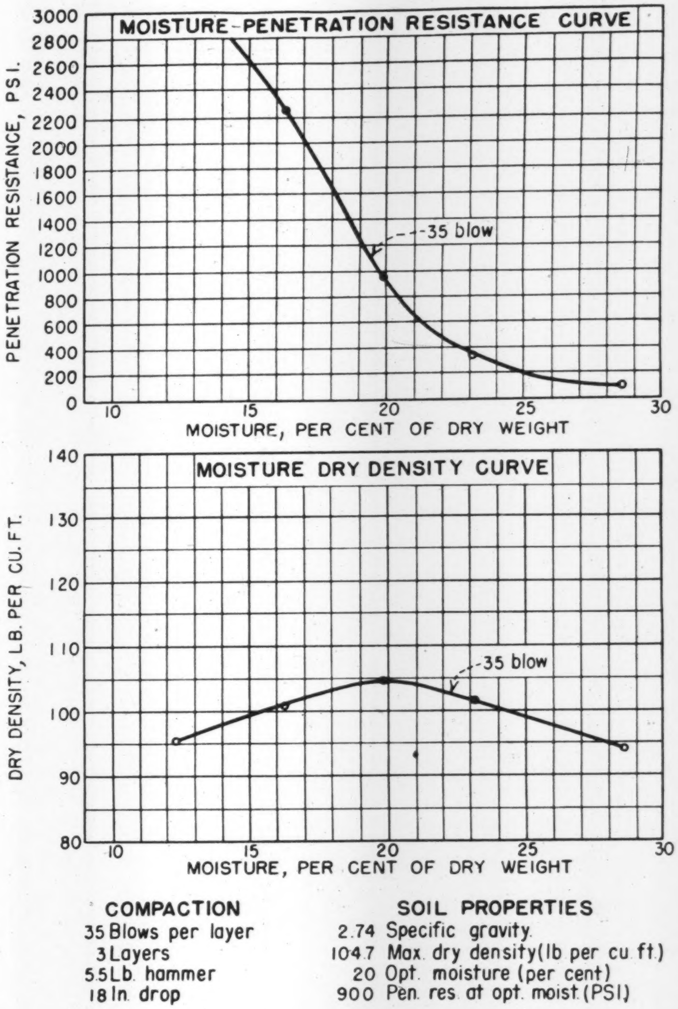

FIG. 7.—Compaction Test Curves.

There are physical limitations of the theoretical 100 per cent density compaction. In Fig. 8, the weight of the rock and the weight of the soil in 1 cu. ft. of soil-rock mixture compacted to theoretical density are shown by the continuous solid line curves which cross below the theoretical density curves. Lines through the unit weight of rock, 103.9 lb. per cu. ft. for 3-in. maximum and 92.3 lb. per cu. ft. for 1-in. maximum, cross the "weight of rock curve" at 74 and 68 per cent rock, respectively. Therefore, physical limitations show that theoretical 100 per cent density cannot be obtained for rock contents above 74 per cent for 3-in. maximum size material and above 68 per cent for 1-in. maximum size material.

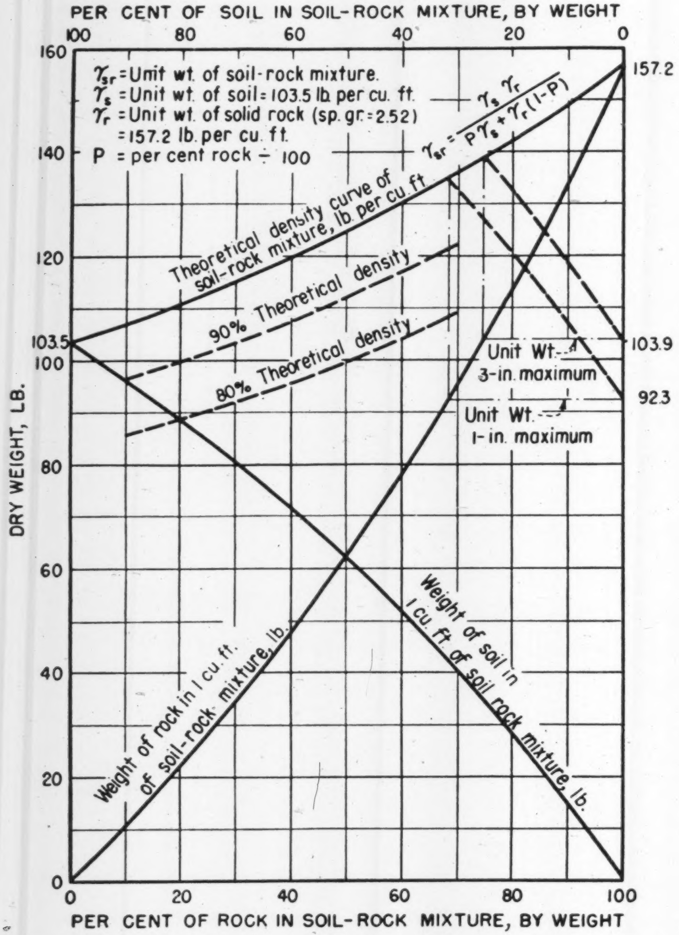

FIG. 8.—Theoretical Placement Densities for Material Containing Rock.

Consolidation Tests:

The consolidations were determined for the various placement conditions, degrees of compaction, and loading pressures in terms of per cent of initial volume. The amount of consolidation before and after saturation and permeability rates which were obtained during the saturation period were determined.

In general, saturation of this material had very little effect on consolidation. The tests which did show some effect of saturation did not show a definite trend, and there is the possibility that much of this additional consolidation from saturation was a result of the loosening up of a local bridging condition that occasionally occurred in the coarser materials. In regard to the pore pressure studies which were subsequently made on the consolidation results, the most critical consideration would be to apply the maximum final consolidation to the pore pressure calculations. Therefore, the pore pressure results which are discussed in the later parts of this paper are based on the final consolidation obtained on all specimens. The consolidation tests results are shown in Table I.

TABLE I.—SUMMARY OF CONSOLIDATION TEST DATA.

Gradation Series	Diameter of Test Cylinder, in.	Degree of Compaction, per cent of Theoretical Density	Rock in Soil-Rock Mixture, per cent	Placement Conditions		Consolidation, per cent of initial volume						Permeability, ft. per yr.		
				Dry density, lb. per cu. ft.	Moisture, per cent	150 psi. Before Saturation	150 psi. After Saturation	100 psi. Before Saturation	100 psi. After Saturation	50 psi. Before Saturation	50 psi. After Saturation	150 psi.	100 psi.	50 psi.
No. 1	8	100	25	113.0	14.5	6.5	6.5	4.7	4.7	3.0	3.4	0.01	0.35	0.30
	8	90	25	102.0	14.5	14.0	14.7	9.4	12.4	1.8	6.8	0.10	0.14	0.75
	8	80	25	90.5	14.5	22.6	23.8	22.0	22.0	16.7	16.7	0.21	0.39	2.43
	20	100	40	120.0	12.4	—	—	2.6	3.9	—	—	—	0.98	—
	20	90	40	108.0	12.4	—	—	7.0	8.5	—	—	—	1.13	—
	20	80	40	96.0	12.4	—	—	15.5	17.3	—	—	—	2.36	—
No. 2	8	100	40	120.0	12.4	2.6	2.6	2.3	2.3	0.5	0.5	0.02	—	0.052
	8	90	40	108.0	12.4	9.2	9.2	6.1	6.1	4.7	4.7	0.18	0.46	11.82
	8	80	40	96.0	12.4	17.1	17.1	11.4	16.9	14.2	14.2	3.44	0.33	298.10
	20	100	56	128.0	10.1	—	—	1.3	1.4	—	—	—	1.46	—
	20	90	56	115.0	10.1	—	—	3.6	5.2	—	—	—	1.60	—
	20	80	56	102.5	10.1	—	—	10.9	13.4	—	—	—	38.95	—
No. 3	8	100	54	127.0	10.4	1.5	1.5	0.8	0.8	0.5	0.5	0.40	0.35	0.05
	8	90	54	114.2	10.4	2.7	4.3	1.3	3.0	0.7	1.0	151.38	27.29	2.94
	8	80	54	101.5	10.4	11.0	14.0	7.9	12.0	2.1	9.7	74.99	29.49	28.13
	20	100	70	136.0	8.1	—	—	0.9	1.0	—	—	—	0.71	—
	20	90	70	122.4	8.1	—	—	1.3	1.5	—	—	—	99.40	—
	20	80	70	109.0	8.1	—	—	6.3	7.7	—	—	—	37.11	—

Permeability Test Results:

The permeability rates obtained are considered to be only approximate because of the short time period available for saturating the specimens and of the piping difficulties which occurred in some of the tests. Many of the specimens were found to be of low permeability. They were generally those representing materials of lower rock contents which were tested under higher applied pressures. There was a tendency for samples having higher rock contents to be pervious. There was also a tendency for the specimens which were compacted at 80 per cent densities to be more pervious than those tested at higher densities. The occurrence of piping is very probable in some of the specimens which showed extremely high permeability rates. In general, the permeability of the material was found to be low except for some specimens of low placement densities with high rock contents which were of greater permeability. The permeability test results are shown in Table I.

Pore Pressure Calculations:

The pore pressure calculations were very similar to those made in placement moisture control investigations of fine material.[5] The following equations are used in the calculations:

$$f = \bar{f} + u \ldots\ldots\ldots (1)$$

where:

f = total stress (fill load),

$\bar{f}$ = effective stress (grain to grain pressure, causing consolidation), and

u = pore pressure.

$$u_c = u_a \left(\frac{V_{ao} + hV_w}{V_{ac} + hV_w} - 1 \right) \quad (2)$$

or

$$u_c = \frac{u_a \Delta}{V_{ao} + hV_w - \Delta} \quad (3)$$

where:

u_c = pore pressure after consolidation (gage pressure),

u_a = initial pore pressure (usually considered as atmospheric pressure—absolute pressure)—since the altitude of the Jackson Gulch Damsite was 7800 ft., u_a in this study was 11.0 psi.

V_{ao} = initial volume of free air in the soil mass in per cent of total initial volume,

V_{ac} = consolidated volume of free air in the soil mass in per cent of total initial volume,

$\Delta = V_{ao} - V_{ac}$ = volume change from consolidation in per cent of total initial volume,

V_w = volume of water in the soil mass in per cent of total initial volume, and

h = capacity of water to dissolve air from Henry's law (approximately 0.02).

Equation 1 shows a simple relation between total stress and effective stress, Eq. 2 is the pore pressure equation shown by L. W. Hamilton,[2] and Eq. 3 is similar to Eq. 2 but is expressed in a slightly different manner for easier use.[3] Equations 2 and 3 are based on Boyle's law for the compressibility of gases and Henry's law for the solubility of air in water at constant temperature. The assumption is made, in using these equations for computing pore pressures, that if a mass containing air, water, and soil solids is loaded without permitting drainage, it will consolidate, and the consolidation occurs as volume change in the air. The air will not only develop pressure according to Boyle's law, but it will dissolve in water according to Henry's law by a weight proportional to the pressure and to the volume of water present.

The one-dimensional consolidation tests, as conducted for this study, allow complete drainage and, therefore provide consolidation data for effective pressures of 50, 100, and 150 psi. From this information, calculations were made to determine the total applied pressures (pore pressure plus effective pressure) necessary to produce the same consolidation that would occur if the soil were sealed. By interpolation between the consolidation data of these *total* pressures, the data for *total* pressures of 50, 100, and 150 psi. were determined.

From the large cylinder test results, only the data for effective pressures of 100 psi. were obtained; and, therefore, the data for total pressures of that amount which require interpolation could not be established. However, the per cent pore pressure has been determined for consolidation data due to the effective pressures for approximate comparisons to the 8-in. cylinder tests.

Pore Pressure Development Curves:

Figures 9, 10, and 11 are graphs showing the consolidation results and the degrees of pore pressures which would develop for assumed sealed conditions. This assumption, which provides the most critical pore pressure interpretation was used in the computations, since many of the specimens were of low permeability. The graphs show the results obtained from tests on material compacted to initial densities of 100, 90, and 80 per cent, respectively of the theoretical density shown in Fig. 8.

The basic grid system of these graphs represents dry density in the vertical direction and per cent rock in the horizontal direction. Superimposed on the scale for per cent rock is a scale of per cent moisture in the total material. These scales are directly related. Since the minus No. 4 material is at a constant moisture of 18 per cent and the plus No. 4 material is considered to have a constant absorption of 3.9 per cent, the change in moisture content of the total material depends only on the change in rock content.

The bottom diagonal line in each figure represents the placement conditions and is obtained from the theoretical curves of Fig. 8. The nearly parallel lines above this placement condition represent the densities which would be obtained for various percentages of consolidation. For example, in Fig. 11, at a rock content of 35 per cent the placement moisture content of the total material will be 13.08 per cent and the

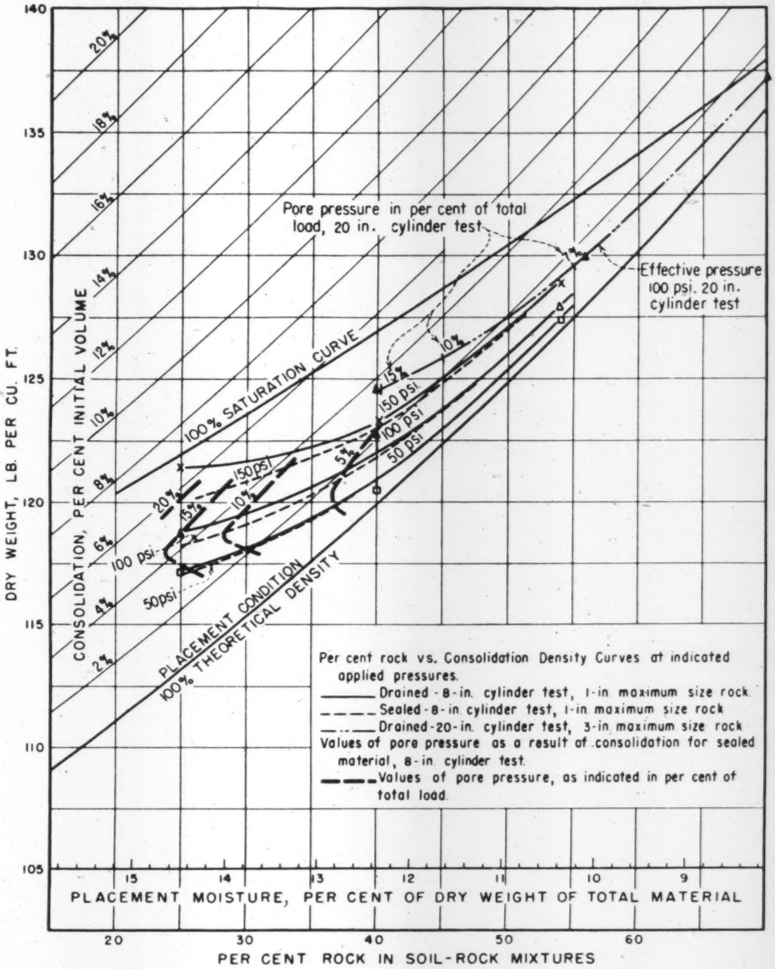

FIG. 9.—Pore Pressure Development Curves 100 per cent Density Placement.

placement dry density, 80 per cent of theoretical, will be 94.0 lb. per cu. ft. When 10 per cent consolidation takes place, the consolidated density will be 104.6 lb. per cu. ft.

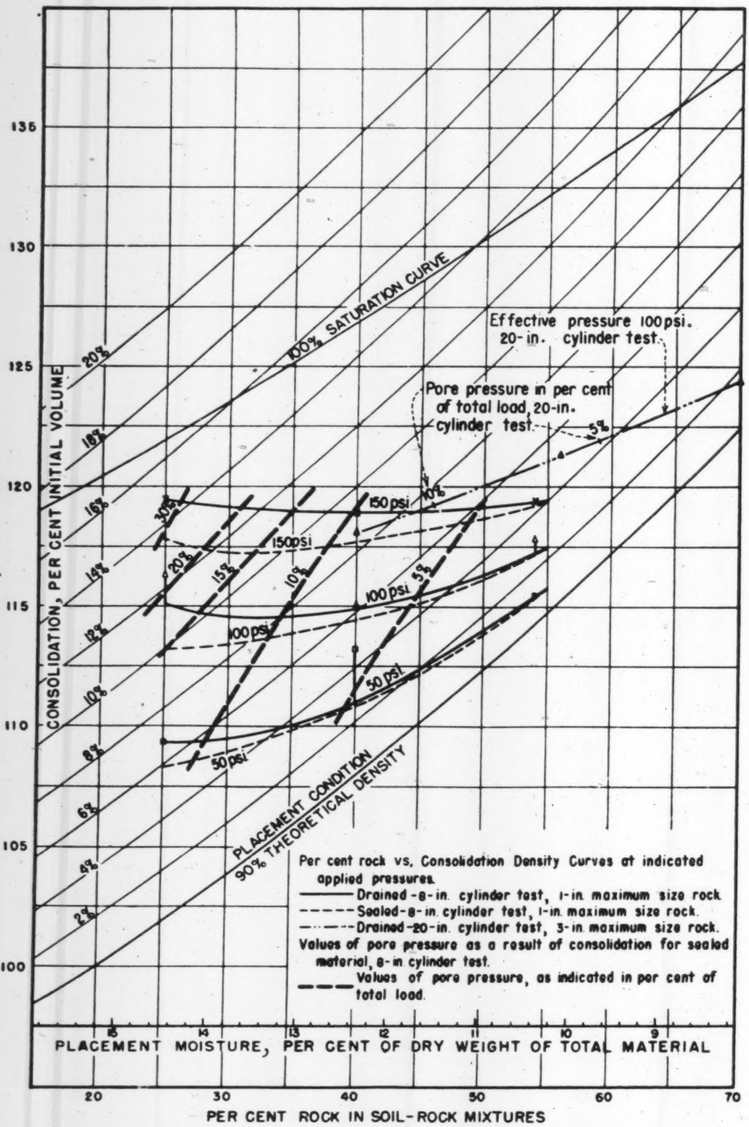

FIG. 10.—Pore-Pressure-Development Curves 90 per cent Density Placement.

In a sealed mass when the consolidation is of such an amount that the air is compressed to a volume which will be totally dissolved by the water, according to Henry's law, the material will be 100

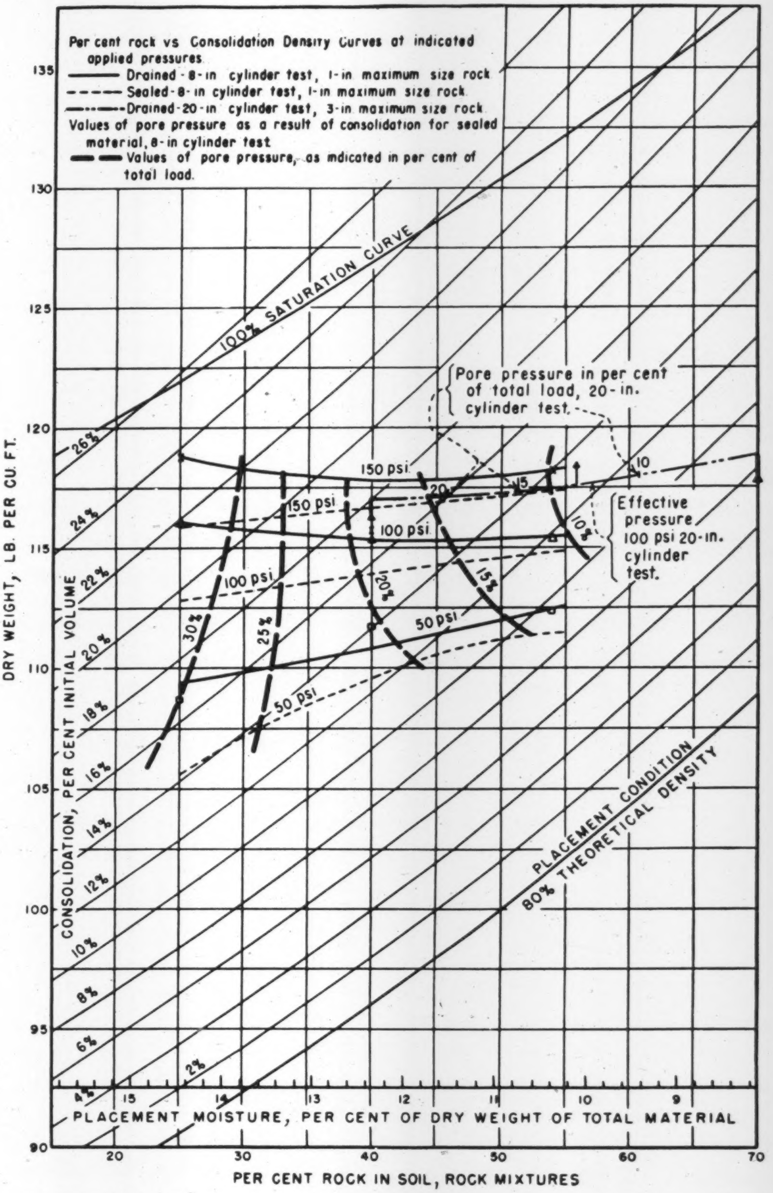

FIG. 11.—Pore Pressure Development Curves 80 per cent Density Placement.

per cent saturated. Percentages of consolidation and consolidated densities at which this will occur are shown for each figure by the heavy lines labeled 100 per cent saturation curve. Furthermore, the pore pressure developed in the sealed mass can be calculated for any point between the placement curve and the 100 per cent saturation curve by the use of Boyle's and Henry's laws. Continuing with the example in the above paragraph, 100 per cent saturation will occur when the sample is consolidated 24.8 per cent to a density of 125.2 lb. per cu. ft.

Superimposed on these graphs are the plottings of the percentages of consolidation which were obtained for the different tests. A series of curves (solid lines) for each graph have been drawn through these plottings and represent the percentage of consolidation and consolidated densities obtained for the different loading pressures of 50, 100, and 150 psi. These curves are labeled in the figures by the loading pressures they represent. Since the test allows complete drainage, the consolidations shown by these curves are for effective (grain to grain) pressures. These curves have been plotted from the results of the 8-in. cylinder tests and are for material of 1-in. maximum size. For example, in Fig. 11, at the rock content of 35 per cent, the curves show that effective pressures of 50, 100, and 150 psi. would cause consolidations of 14.6, 18.6, and 20.2 per cent and consolidated densities of 110.3, 115.5, and 118.0 lb. per cu. ft., respectively. The consolidation results of the 3-in. maximum material are shown for the effective pressure of 100 psi. by the dash-dot line labeled 20-in. cylinder tests.

By combining the effective consolidation pressures of 50, 100, and 150 psi. with the pore pressures that would be developed under sealed conditions for the respective consolidations, the total pressures necessary to produce the same consolidation of the material under sealed conditions are obtained. Then, by interpolation, the consolidation percentages can be determined for total pressures of 50, 100, and 150 psi. These are shown in the graphs by dashed lines labeled with their respective total pressures. For the same rock content as shown above, 35 per cent in Fig. 11, the curves show that *total pressures* of 50, 100, and 150 psi. would cause consolidations of 13.3 17.1., and 19.1 per cent and consolidated densities of 108.5, 113.5, and 116.4 lb. per cu. ft., respectively. The total pressure line could not be drawn for the large 20-in. cylinder test results because the single effective pressure line of 100 psi. did not provide enough data for interpolation.

Pore pressures, in per cent of total load, are shown by the heavy dashed lines which cross the total pressure (thin dashed) lines. Also, the pore pressures in per cent of total load are indicated along the effective 100 psi. pressure line for the 20-in. cylinder test results.

The actions of consolidation and corresponding pore pressure development in soil-rock mixtures assuming sealed conditions may be demonstrated by the diagrammatic drawings of Fig. 12. These drawings have been made to scale for rock contents of 30, 40, and 50 per cent from the test results shown in Figs. 9, 10, and 11. For each of the placement conditions the total of the non-compressible volumes, which include rock solids, soil solids, and water, and the compressible volumes, which include the free air and the dissolved air (2 per cent of the water volume), are shown. As the rock content increases, the pore pressure developed by consolidation decreases considerably. For example, at 80 per cent density placement and 100 psi. loading, the developed pore pressures are 28 per cent for 30 per cent rock, 19 per cent for

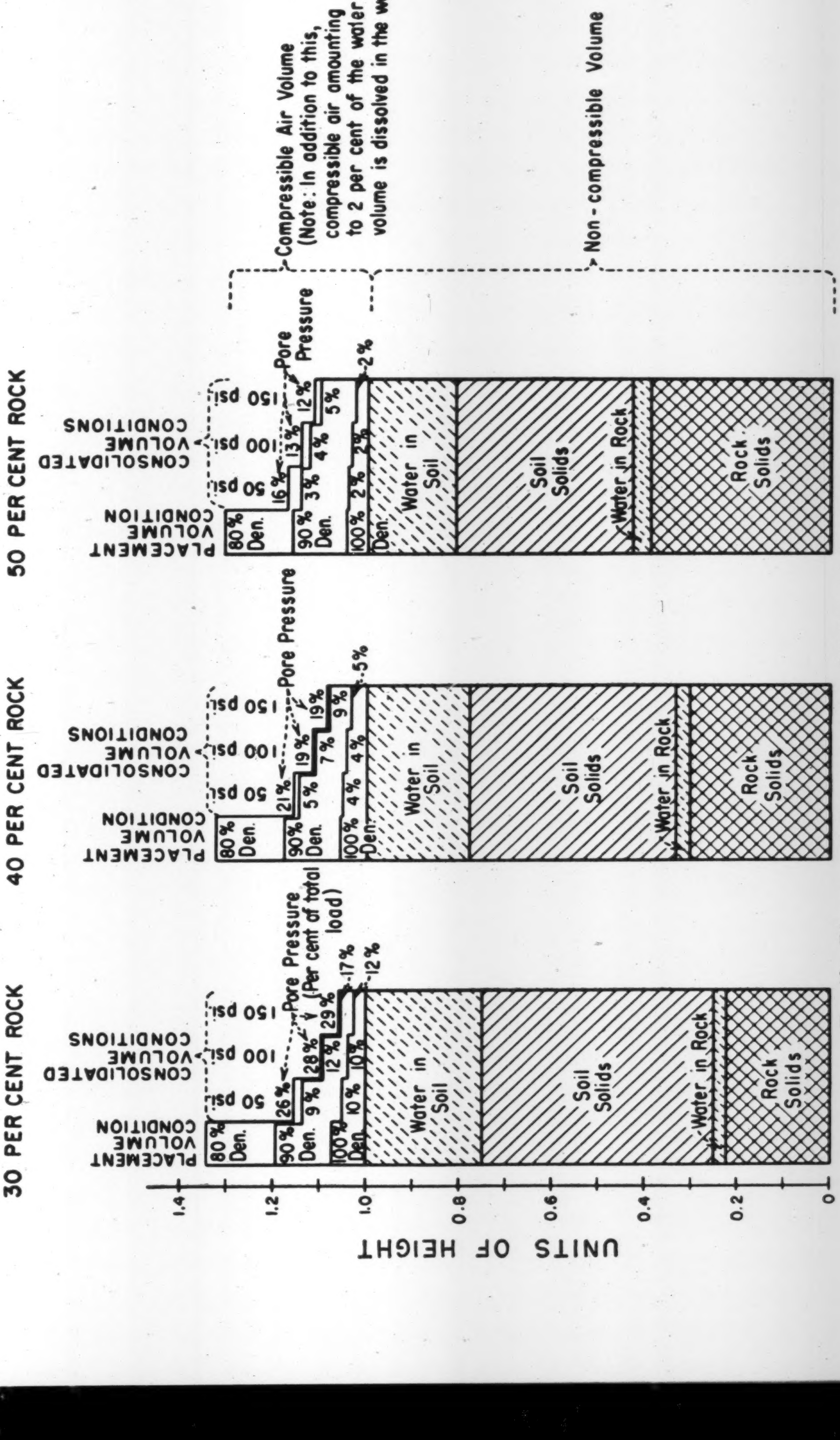
30 PER CENT ROCK
40 PER CENT ROCK
50 PER CENT ROCK
PLACEMENT VOLUME CONDITION
CONSOLIDATED VOLUME CONDITIONS
50 psi
100 psi
150 psi
80% Den.
90% Den.
100% Den.
Pore Pressure (Per cent of total load)
Pore Pressure
26%
28%
29%
9%
12%
10%
10%
17%
12%
21%
19%
19%
5%
7%
9%
4%
4%
5%
16%
13%
12%
3%
4%
5%
2%
2%
2%
Water in Soil
Soil Solids
Water in Rock
Rock Solids
Compressible Air Volume (Note: In addition to this, compressible air amounting to 2 per cent of the water volume is dissolved in the water.)
Non-compressible Volume
UNITS OF HEIGHT
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0

FIG. 12.—Diagrammatic Drawings of Consolidation and Corresponding Pore Pressure Development.

40 per cent rock, and 13 per cent for 50 per cent rock. The reduction in pore pressure as rock content increases is caused by reduction in consolidation. It is important to note that the reduction in consolidation is more than can be accounted for by the displacement of compressible soil by solid rock and that particle interference of the rock is an important factor in reducing the consolidation of the total material.

TABLE II.—MAXIMUM PROBABLE PORE PRESSURE DEVELOPED IN THE SOIL-ROCK MIXTURES, ASSUMING SEALED CONDITIONS.

Degree of Compaction, per cent of theoretical density	Pore Pressure, per cent of total load (Range of pore pressure for respective range of rock content)		
	150 psi. Total Load	100 psi. Total Load	50 psi. Total Load
1-in. maximum size rock—Range of rock content, 25 to 55 per cent[a]			
100	20 to 0	14 to 0	16 to 0
90	29 to 3	15 to 2	12 to 2
80	36 to 9	33 to 11	28 to 13
3-in. maximum size rock—Range of rock content, 40 to 70 per cent[a]			
100		15 to 6	
90		12 to 2	
80		25 to 5	

[a] The ranges of rock content shown are those used in testing materials of the respective maximum sizes. Comparisons of materials for the various rock contents are shown in Tables III and IV.

The drawings of Fig. 12 further demonstrate that the material will have a greater probability of developing pore pressures when stressed if it is placed at low degrees of compaction. For example, at 30 per cent rock content and 100 psi. loading, 80 per cent density material developed 28 per cent pore pressure; 90 per cent density material, 12 per cent pore pressure; and 100 per cent density material, 10 per cent pore pressure. The reason for this is that the proportion of initial air volume to consolidated air volume is greater in the 80 per cent density material than in the materials at the higher placement densities. Considering only the free air volume, the relation discussed above may be observed in Fig. 12. For example, at 30 per cent rock and 100 psi. loading, 80 per cent density material with an initial free air volume of 0.34 has a consolidated air volume of 0.10 and 90 per cent density material with an initial free air volume of 0.19 has a consolidated air volume of 0.09. The proportion of initial volume to consolidated volume is greater for the 80 per cent density material $\left(\frac{0.34}{0.10} = 3.40\right)$ than for the 90 per cent density material $\left(\frac{0.19}{0.09} = 2.11\right)$. This relation may be readily observed in Eq. 2.

The above pore pressure conditions may be relieved somewhat because of higher permeability tendencies for the lower degrees of compaction; but, since most of the permeability values were still quite low, the most critical pore pressure condition would be for the assumption that material is sealed.

The consolidation and pore pressure results, for comparing the effects of the different testing conditions, are summarized in Tables II, III, and IV. The conclusions which follow are based on the results shown in these tables and in Fig. 12. Table II summarizes the pore pressure values in per cent of total load for the ranges of rock contents studied and shows the effect of various loading pressures and various degrees of compaction. The consolidation and pore pressure data for 1-in. and 3-in. maximum size materials are compared in Tables III and IV, respectively. They are arranged for comparisons by gradation series or rock content. The pore pressure values, shown for the 3-in. maximum size material, are from the effective 100-psi. pressure line, since the total pressure line could not be drawn. However, based on the observations of the 8-in. cylinder tests, the ranges of values are reasonably accurate for the purpose of comparison.

TABLE III.—CONSOLIDATIONS FOR 100 PSI. EFFECTIVE PRESSURE AT VARIOUS ROCK CONTENTS FOR COMPARING EFFECT OF GRADATIONS AND MAXIMUM SIZES OF ROCK.

Rock Content, per cent	Consolidation, per cent of initial volume								
	100-per cent Density			90-per cent Density			80-per cent Density		
	1-in. maximum size	3-in. maximum size	Gradation	1-in. maximum size	3-in. maximum size	Gradation	1-in. maximum size	3-in. maximum size	Gradation
25	4.6			11.5			21.8		
40	1.5	3.5		6.1	8.6		16.7	18.0	
55	0.7	1.5	Series I	2.5	5.3	Series I	11.5	13.0	Series I
70		1.0	Series II		1.5	Series II		8.5	Series II
			Series III			Series III			Series III

TABLE IV.—MAXIMUM PROBABLE PORE PRESSURE DEVELOPED IN THE SOIL-ROCK MIXTURES, FOR 100 PSI. LOAD ASSUMING SEALED CONDITIONS, FOR COMPARING EFFECT OF GRADATIONS AND MAXIMUM SIZES OF ROCK.

Rock Content, per cent	Pore Pressure, per cent of total load								
	100-per cent Density			90-per cent Density			80-per cent Density		
	1-in. maximum size	3-in. maximum size	Gradation	1-in. maximum size	3-in. maximum size	Gradation	1-in. maximum size	3-in. maximum size	Gradation
25	14			15			33		
40	5	15		7	12		19	25	
55	0	7	Series I	2	7	Series I	11	13	Series I
70		6	Series II		2	Series II		5	Series II
			Series III			Series III			Series III

CONCLUSIONS

1. The testing procedure discussed herein proved applicable for demonstrating the actions of consolidation and pore pressure development in soil-rock mixtures. The test is perhaps too long to be standardized for the analysis of all soil-rock mixtures but is useful for similar special studies. However, the generalized information gained from this study has application to all soil-rock mixtures.

2. The probability of high pore pressure decreases considerably as the amount of rock is increased. This is true, as shown by the test results, because the consolidation of the total material is restricted as the rock content becomes greater. The restriction of consolidation is greater than can be accounted for by the solid rock volume displacing compressible soil.

3. The calculated pore pressures for the sealed conditions under load were considerably higher for material placed at 80 per cent density than at 90 and 100 per cent density. This was caused by the large quantities of consolidation for the material at 80 per cent density, in spite of the fact that this material was less saturated and had greater compressibility in the pore fluid. This pore pressure

condition might be relieved somewhat by the probable greater permeability of the material at 80 per cent density. However, the permeability rates were low for some specimens, and a most critical pore pressure study would be to assume the sealed condition.

4. When the rock contents were 25 per cent and greater and applied pressures were 150 psi. or less, the pore pressure of this material was found to be not greater than 36 per cent. This value is based on a moisture content of 18 per cent for the fine material and an absorption of 3.9 per cent for the rock.

5. The comparisons of tests on the 1-in. and 3-in. maximum size mixtures indicate the following:

(*a*) *For each gradation series*, the 3-in. maximum size material showed less consolidation and either the same or less pore pressure than the 1-in. maximum size. This is mainly attributed to the fact that the higher rock content for the 3-in. maximum material caused a decreased consolidation. Although the moisture content was decreased as rock content increased, the greater densities actually decreased the compressible volume of air and this would have tendencies toward higher pore pressure. Therefore, since the test results showed that the pore pressure for the 3-in. maximum material was the same or less than for 1-in. maximum material, the decrease in consolidation for higher rock content had the greatest influence on the pore pressure calculations.

(*b*) *For equal total rock contents*, the 3-in. maximum size material showed greater consolidations and greater pore pressures than the 1-in. maximum size. This may be attributed to the fact that the 3-in. maximum size material is better graded and more adaptable to a dense arrangement of particles than the 1-in. maximum size. That is, the 1-in. maximum size was more uniform in its coarse sizes and was probably subject to more particle interference.

Acknowledgments:

The author thanks Mr. W. G. Holtz for presenting the paper at the A.S.T.-M. Pacific Area National Meeting, and Mr. Holtz and Mr. A. A. Wagner for their helpful suggestions and criticisms in preparation of the paper.

DISCUSSION

Mr. W. J. Turnbull[1] *(presented in written form)*.—Mr. Gibbs has selected a very important phase of soils testing for his practical research investigation. A greater knowledge of the effects of pore pressure on the strength of soils is certainly needed. In particular, the demonstration that pore pressures can be developed in such basically stable materials as the soil-rock mixtures is considered very important. The development of the method used by Mr. Gibbs and by other Bureau of Reclamation engineers is believed to be an important step forward. The lack of knowledge on the actual pore pressures developed in prototype structures is a gap which must be bridged before it can be said that we are using principles of soil mechanics in rational design procedures. In numerous instances, soils engineers are somewhat deluding themselves that they are using rational design even though the information on pore pressures in the laboratory test and in the prototype is sketchy or completely lacking.

The study by Mr. Gibbs on a specific soil mixture covering ranges in amounts of the coarse material as well as a range in degree of compaction furnishes, at the least, valuable qualitative information. This study demonstrates and accentuates the great need for detailed correlation between the laboratory and the field so that more information of a quantitative nature is forthcoming. With laboratory-field correlation, the test procedure followed by Mr. Gibbs would have great value as a laboratory test, in spite of the difficulty and expense of conducting it. This difficulty and expense would readily be offset by the knowledge gained. It is the opinion of the writer that in too many cases the soils engineering profession is striving for simple, empirical tests. This is certainly all to the good as far as accomplishing specific projects; however, the need for basic investigations is very apparent, and any type of laboratory test which can be conducted to furnish basic information on pore pressure and its correlation from laboratory to prototype can be readily justified.

Mr. R. G. Hennes.[2]—I wonder how the author's data might have looked had pore pressures been expressed as functions of the actual pressure on the matrix, or filler material, rather than as percentages of total load.

When the coarse aggregate constitutes the greater part of the total volume of the fill, it may be assumed that the interlocking rock fragments form a relatively stiff framework or skeleton, which will carry a disproportionately large portion of the imposed load, while the sheltered filler material remains unconsolidated.

That sort of situation is not uncommon in natural soil deposits. Recent samples from a mill site on Vancouver Island were characterized by clay-filled pores in rather coarse gravel. The water contents of this clay were much higher than the values corresponding to the weight of the overburden despite the probability that time and drainage conditions were adequate to permit for complete consolidation. Probably consolidation really was substantially complete under the lesser percentage of overburden pressure which was actually transmitted to the filler material. The rest of the overburden was carried by the relatively incompressible gravel.

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[1] Chief, Soils Div., Waterways Experiment Station, Corps of Engineers, Vicksburg, Miss.

[2] Professor of Civil Engineering, University of Washington, Seattle, Wash.

The author stated that the decrease in consolidation with increasing rock content was more than could be accounted for by the displacement of soil by rock. If the rock carried relatively more load than its proportionate part of the total volume, it did so only because the rock structure was stiffer than the interspersed clays.

Data on stress in the matrix would form an interesting supplement to this valuable paper.

MR. W. G. HOLTZ.[3]—In this particular dam, as in the designing of most dams, certain assumptions were made as to the behavior of the materials being used. In this case two assumptions were of primary importance: (1) The permeability of the clayey soil-gravel material, and (2) The consolidation and pore pressure characteristics of the material. The results of the tests contained in Mr. Gibbs' paper supported the assumptions made; that is, (1) the permeability of the material would not be extremely low, with the relatively high rock contents of this material and, because of the drainage thus obtained, there would be less likelihood of high total pore pressures developing during construction; and, (2) the consolidation, and accompanying pore pressure development, would not be critical because of the high rock content.

I should like to emphasize further at this time what Mr. Turnbull has said with regards to the need for more information to bridge the gap between laboratory tests and actual field performance. However, I should like to make this point clear with respect to the actual measurement of pore pressures in our earth structures. The Bureau of Reclamation has built quite a number of large earth dams during the past several years. Since about 1940 we have installed a large number of settlement and pore pressure measuring instruments in these structures. The results of some of these measurements are described in the article by F. C. Walker and W. W. Daehn, Proceedings of the Second International Conference on Soil Mechanics and Foundation Engineering, (footnote 4 of paper). From these measurements we have worked out correlations between the field pore pressure and consolidation values and the values secured in the laboratory, and in many cases have worked out limiting placement moisture controls to reduce construction pore pressures.[4]

MR. H. J. GIBBS (*author's closure by letter*).—The author welcomes Mr. Turnbull's comments regarding the need for more correlation between field and laboratory testing as this is something needed in all phases of soil mechanics. Foundation and embankments are designed and constructed on the basis of theoretical assumptions, and too frequently the observations of the action of the prototype are neglected. In answer to this Mr. W. G. Holtz mentioned the work which the Bureau of Reclamation has done in observing consolidation and pore pressure conditions which exist in actual structures. In addition to the reference given by Mr. Holtz, I would like to mention another paper titled "Comparison Between Laboratory Test Results and Behavior of Completed Embankments and Foundations" by F. C. Walker and W. G. Holtz, presented at the 1950 Spring Meeting of the American Society of Civil Engineers, Los Angeles, Calif., April, 1950.

The author wishes to present the following views in answer to Mr. Hennes' discussion. For a certain soil-rock condition having low permeability and having certain placement density and moisture, the pore pressure is dependent only on the volume change. Since the rock particles are not considered to have volume change, the consolidation occurs in the soil matrix. In the paper, the pore pressures (in unit pressure dimensions) were determined from the volume changes in the total material resulting from applied load. Correlative with this, for the above reason, the same pore pressure would result if volume changes in the soil matrix only were considered. However, it is general practice in the Bureau to report pore pressure in per cent of total applied load since this indicates the portion of the load being carried by the pore fluid in comparison to the portion being carried by solid material, whether it be soil-rock or just soil. Therefore, if the pore pressures were stated as a function of the pressure occurring in the soil matrix, the portion of the load carried by the soil-rock structure would not be justly represented. The fact that the rock structure in case of high rock content carries a large portion of load is a valuable item in reducing pore pressure, as consolidations are reduced a greater amount than that accounted for by displacement of compressible soil by relatively incompressible solid rock.

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[3] Head, Earth Materials Lab., U. S. Bureau of Reclamation, Denver Federal Center, Denver, Colo.

[4] W. G. Holtz, "The Determination of Limits for the Control of Placement Moisture in High Rolled Earth Dams," *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 1240 (1948).

The example of the mill site on Vancouver Island stated by Mr. Hennes demonstrates the ability of the rock structure in reducing consolidation (probably by bridging of rock particles) and reducing pore pressure. The water content of the clay matrix was greater than that which would result after complete consolidation of the clay if the total overburden pressure had been exerted on it alone. Therefore, the author is in agreement that the incompressible rock was carrying a large portion of the overburden and reducing the overburden carried by the clay matrix.

ELECTRICAL RESISTIVITY METHOD APPLIED TO THE INVESTIGATION OF CONSTRUCTION MATERIALS DEPOSITS*

By E. A. Abdun-Nur[1] and Dart Wantland[1]

Synopsis

The problem of investigating deposits of rock and gravel in the plains area (the Dakotas, Nebraska, Kansas, Oklahoma, and the eastern portions of Montana, Wyoming, and Colorado) is outlined. The electrical resistivity method as applied to the solution of this problem by the Bureau of Reclamation is discussed. Techniques and simplified apparatus are described (Appendix). The usefulness of the resistivity method in supplementing and guiding drilling in the exploration of such deposits is demonstrated by examples of actual investigations. Cost data are presented. It is concluded that in deposits of similar materials, the electrical resistivity method provides an effective, quick, and economical means of determining depths, delineating the extent, and estimating the quantities of materials involved.

As a general rule, rock deposits and sand and gravel deposits for use as construction materials are not difficult to find in most parts of the United States and do not very often present unusual problems in their investigation and exploitation. A notable exception to this is found in the plains region, comprising the Dakotas, Nebraska, Kansas, Oklahoma, and the eastern portions of Montana, Wyoming, and Colorado. In this large and fairly level area, hard rock and gravel deposits near the surface of the ground are extremely scarce. When they occur, they are usually of marginal quality and variable in hardness, thickness, and extent. Often such materials have to be shipped in for hundreds of miles from outside this large area; but, occasionally, a deposit is found which offers promise of being useful on an adjacent project, thus saving the high cost of importing materials.

The variable character of these deposits makes an evaluation of the variation in quality, the mapping, and the estimating of quantities of useful and waste material very difficult and costly. Drilling such deposits to obtain complete information is out of the question, inasmuch as there is no assurance that the condition found at any given drill hole is valid even as little as 10 ft. away. Faced with such a problem, it was decided to try the electrical resistivity method of geophysical investigation in an attempt to determine the variations in thickness and lateral extent of such deposits, thus reducing the amount of drilling and permitting the location of drill holes where they would supply the most information. The drill holes, in turn, would then supply the control for the geophysical measurements and provide core samples that would permit an

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* Presented at the Session on Soils held at the First Pacific Area National Meeting of the Society, San Francisco, Calif., October 10-14, 1949.

[1] Engineer, Materials Investigations and Field Research, and Geophysicist, Geology Section, respectively, Bureau of Reclamation, Denver, Colo.

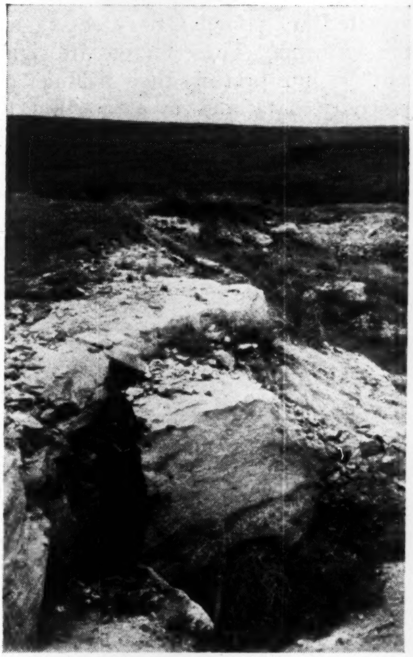

FIG. 1.—Ludell Quarry Outcrop, Rawlins County, Kans. Massive Bed of Hard Silicified Sandstone. Extent of Deposit Not Visible from Surface Examination.

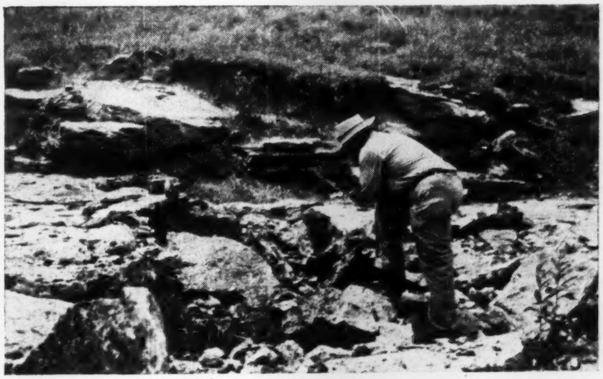

FIG. 2.—Herndon Quarry Outcrop, Rawlins County, Kans. Comparatively Soft Silicified Sandstone Illustrating Discontinuous and Variable Character of Rock and Heavy Overburden. Extent of Deposit Not Visible from Surface Examination.

evaluation of the quality of the material involved. In the case of gravel deposits, test pits are substituted for drill holes. As far as is known to the authors, this method has not been used previously in the exploration of this type of rock deposits, and it is hoped that the discussion and data presented in this paper may be useful to other engineers engaged in similar work.

MATERIALS AND METHODS

Most of the marginal rock deposits, referred to above, are composed of what is locally known as "quartzite," although petrographically, they are silicified sandstones which vary in hardness. In some instances, these are sufficiently hard to be considered as potential sources of riprap or crushed blanket materials in the construction of dams and other irrigation structures. The rock ranges, as a rule, from very soft sandstone to hard silicified material. In profile, the thickness varies considerably and changes rather rapidly from a usual maximum of possibly 8 to 10 ft. to as little as 2 or 3 in. This variation is also in evidence laterally as the rock narrows down and pinches out rather frequently and, at other times, covers extensive areas. In most instances, the rock has visible outcrops, but is usually covered with a comparatively heavy overburden within a very short distance from the face (Figs. 1 and 2). In fact, the thickness of the overburden is frequently greater than the thickness of the rock. This may be, at times, an important economic consideration.

The origin of these silicified sandstone deposits, which are fairly common in western and central Kansas and adjacent parts of Nebraska and Colorado, is not certain (**1**).[2] There is evidence that the silicified rock deposits are remnants of material laid in old braided stream channels. The sinuous course of such deposits, evident in many cases (Fig. 3), coupled with the fact that they appear to be lenticular in cross-section with sharp boundaries (as they would be if deposited in channels), likewise suggests such an origin. These factors are significant in interpreting the drilling and resistivity data and in estimating the available yardage of rock, as continuity of the desposit laterally or in thickness cannot be safely assumed except over short distances or between closely spaced control points.

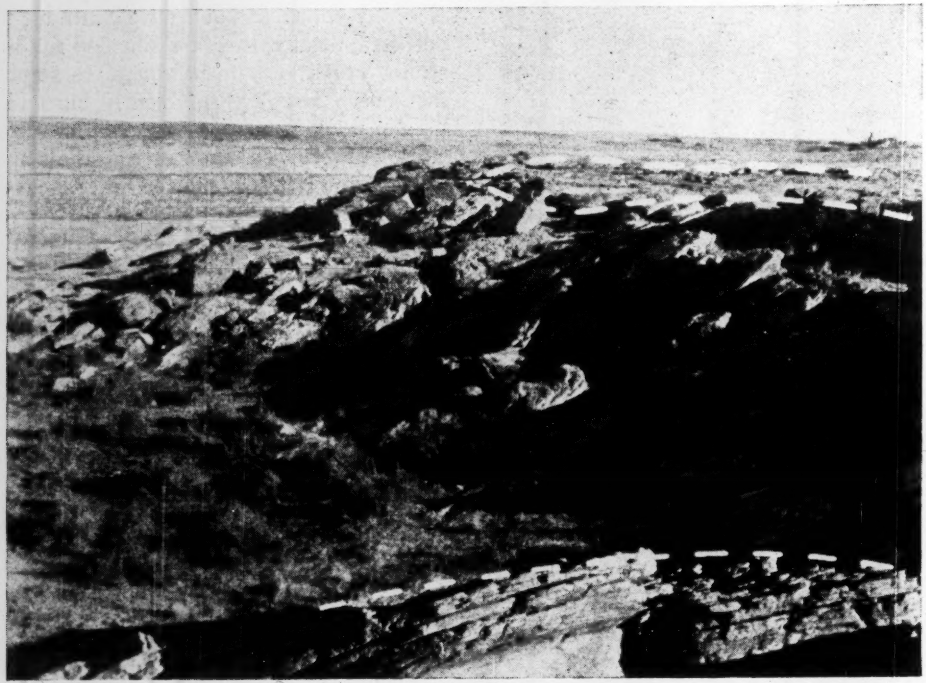

FIG. 3.—"Quartzite" Deposit "D" Near Akron, Washington County, Colo. Dotted Line Shows Sinous Nature of Outcrop.

The resistivity method, which is known to many engineers, uses as its basis the fact that the electrical resistance or resistivity of different natural materials (clays, sands, gravels, shales, various types of rocks, etc.) is a physical property which characterizes them almost as definitely as their density. The measurement of the resistivity of such materials, in their natural state, affords

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1378.

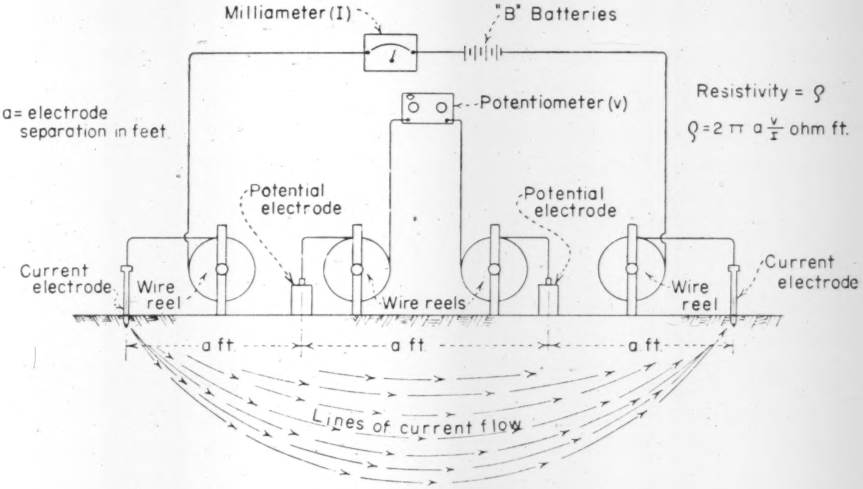
Milliameter (I)
"B" Batteries
Potentiometer (v)
Resistivity = ρ
$\rho = 2\pi a \frac{v}{I}$ ohm ft.
a= electrode separation in feet
Potential electrode
Potential electrode
Current electrode
Wire reel
Wire reels
Wire reel
Current electrode
a ft
a ft
a ft
Lines of current flow

FIG. 4.—Diagrammatic Arrangement of Field Resistivity Equipment.

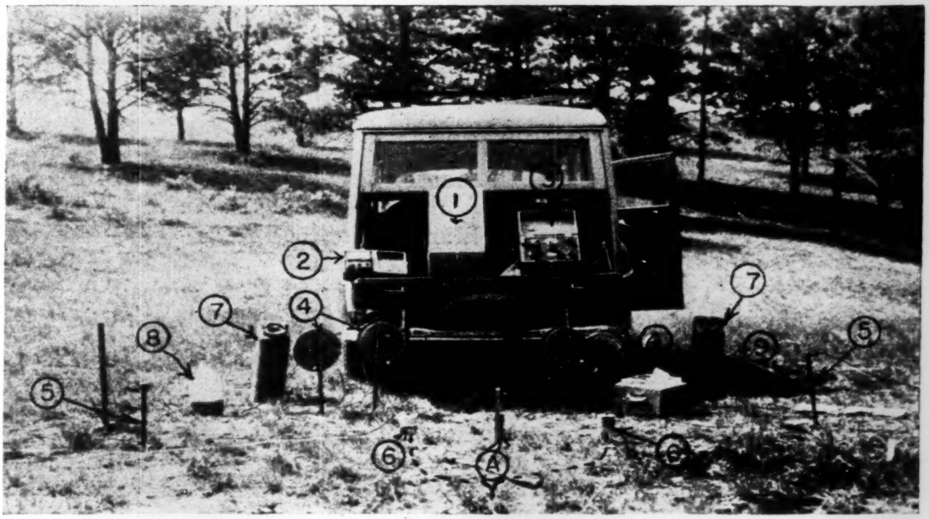
1
2
3
4
5
5
6
6
7
7
8
A
"a"

FIG. 5.—Equipment for Making Field Electrical Resistivity Measurements.

1. Battery-Power source
2. Milliammeter
3. Potentiometer
4. Reels
5. Field electrodes
6. Porous pots
7. Water cans
8. Copper sulfate solution
9. Box for porous pots

"a" spacing in feet

A. Center of spread

therefore an opportunity for distinguishing one type from another without making a physical excavation to expose the materials in question. The resistivity method is, in effect, a field application of the well-known Ohm's law and depends on the relationship of current, resistance, and voltage. It consists in passing and measuring an electrical current through the ground by means of two current electrodes. At the same time, the voltage difference is measured at two other points located between the current electrodes. The arrangement of the apparatus for field measurements is shown diagrammetrically in Fig. 4. Figure 5 is a photograph of the actual equipment. The relationships of the voltage, the current, and the distance separating the electrodes permit a calculation of the resistivity. The volume of material through which the current passes is proportional to the distance between the electrodes. Therefore, the depth of the material included in the measurement is proportional to the distance between the electrodes. It has been found that the effective depth of measurement is roughly of the same order as the electrode separation. The application of this method and the simplified equipment adopted by the Bureau of Reclamation are described in detail in the Appendix.

The electrical resistivity method may be used to make: (*a*) resistivity depth measurements at a selected point, or (*b*) fixed-depth resistivity traverses along a given line. In the resistivity depth measurement, the electrode spacing is progressively increased to pick up changes in resistivity with depth at a point and may be likened to "electrical drilling." The fixed-depth resistivity traverse in which the electrodes are maintained at a fixed spacing while being moved along a line, thus indicating the lateral changes in materials, as contrasted with the vertical changes in the resistivity depth measurement, may be likened to "electrical trenching."

APPLICATIONS OF THE RESISTIVITY METHOD TO THE INVESTIGATION OF ROCK DEPOSITS

Fremont Butte "Quartzite" Deposit (Colo.):

The first trial of the resistivity method by the Bureau of Reclamation, for the purposes described in this paper, was made at Fremont Butte, eight miles northwest of Akron, in northeastern Colorado, in April, 1948. Drilling at this location had indicated the variable nature of the material, and it had become apparent that to obtain reliable information solely by drilling would be very costly.

The purpose of this work was: (*a*) to use the existing drill holes as control for evaluating the resistivity method in its application to the determination of depth, thickness, and extent of silicified sandstone; (*b*) to map the deposit and estimate the quantity of available rock; (*c*) to determine the speed and cost of resistivity work as compared to drilling in the exploration of similar bodies of rock.

This deposit consists of a narrow sinuous outcrop of rock extending for a distance of about 1½ miles in a broad arc running roughly in a north and south direction. The hard rock varies in thickness from 1½ ft. to over 20 ft. in some places and in width from 25 to 150 ft. or more.

The field program consisted: (*a*) in making resistivity depth measurements at the existing nine diamond-drill holes in order to determine the thickness and depths of the siliceous sandstone and to compare these results with the drill logs of the holes; and (*b*) in running fixed-depth resistivity traverse lines between three pairs of drill holes to check the location of the edges of the rock layer, as shown by drilling.

From resistivity depth measurements, it was found that where siliceous sandstone was included in the volume of material being measured the resistivity was comparatively high and ranged from about 400 to 700 ohm ft. (Fig. 6). Where no such hard rock was present, the values of the resistivity were low, as is characteristic of ordinary soils composed of clay, sand, or shale, the values ranging between 50 and 200 ohm ft. (Fig. 7).

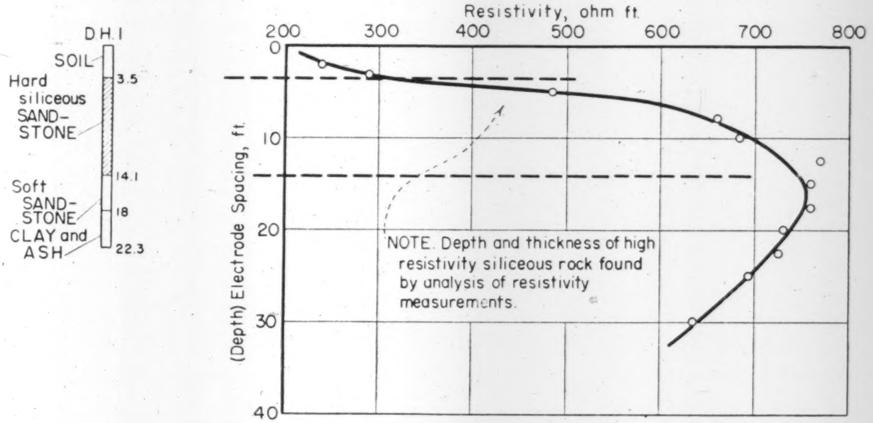

Fig. 6.—Resistivity Depth Curve Indicating Presence of High Resistance Siliceous Sandston Fremont Butte, Colo., April 7, 1948.

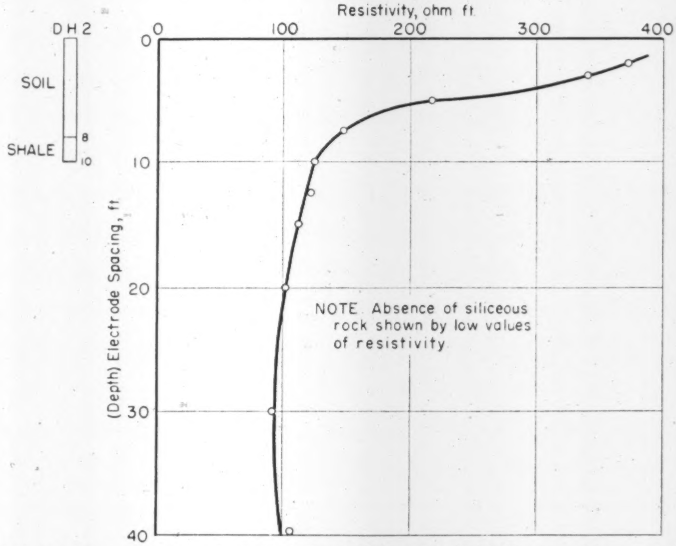

Fig. 7.—Resistivity Depth Curve No High-Resistivity Rock Indicated, Fremont Butte, Colo. April 7, 1948.

The fixed-depth resistivity traverse lines were run back, from the edge of the rock exposure, across the area where the rock was covered by overburden. As evidenced by Fig. 8, which shows the results of one of the traverses, it was possible to locate the edge of the silicified rock rather accurately.

The exposed rock ledges, supplemented by the drill logs, permitted the interpretation of the resistivity results to be made, by inspection, on the basis of the appearance of the plotted resistivity depth curves and fixed-spacing resistivity traverses. This proved satisfactory for the purpose without using one of the more exact analytical interpretation methods referred to in the Appendix. Lacking the drilling control that was available, such visual interpretation might not have been feasible.

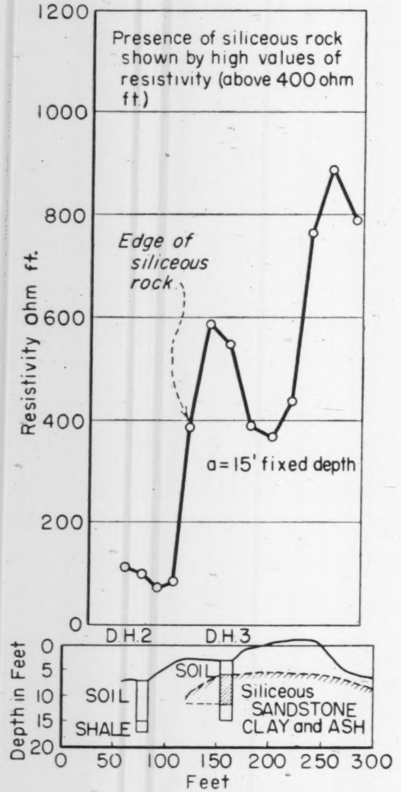

FIG. 8.—Comparison of Data from Resistivity Traverse and Drilling for the Purpose of Locating Edge of Rock.

The work in this area was later extended to cover most of the deposit. Check drilling, which was materially reduced from the amount that would have otherwise been required and which was spotted at strategic points on the basis of the electrical resistivity data, confirmed the geophysical results and demonstrated their value.

Under the conditions existing at the Fremont Butte rock deposit, the resistivity depth curves, with a minimum of drilling for control, gave conclusive evidence of the presence or absence of rock, and its depth, thickness, extent, and variation. In addition, the changes in resistivity gave a qualitative indication of the variation in the quality of the rock. In general, it was found that the higher the resistivity the better the quality of the silicified sandstone. It is hoped that laboratory research will permit more reliable correlations on quality in the future.

In eight days, in the field at Fremont Butte, 13 resistivity depth curves and seven lines of fixed spacing traverse were completed by a four-man crew. It was found that such a crew could run four resistivity depth curves to a maximum electrode spacing of 50 ft. in an 8-hr. day. One line of fixed depth traverse, 150 ft. long, takes approximately the same time to complete as one resistivity depth curve. Thus, the 13 depth curves and seven resistivity traverse lines as set up comprise 20 units of work.

The field conditions at Fremont Butte and at most similar deposits in the plains area are rather favorable, inasmuch as the ground is quite level and very few obstructions occur. Surveying to locate resistivity points requires the same order of accuracy as surveying to locate drill holes.

The cost of this work, for a four-man party, is estimated to be approximately

$2500 per month. The personnel for resistivity field crews do not have to be highly trained and they are usually picked by the geophysicist, under whose supervision they work, from available subprofessional men on the staffs of the various projects. Assuming 22 work days in each month and four units of resistivity work per day, the cost per unit of work is placed at approximately $30. This provides information to a depth of approximately 50 ft. at roughly 60 cents per foot of depth. Current Bureau of Reclamation figures indicate that the cost of manning and operating a core-drill is $75 per 8-hr. shift. In deposits of this kind, it is possible to drill one core hole to a depth of 50 ft. per day under ordinary conditions, which gives a cost of $1.50 per ft. It is thus evident that "electrical drilling" is much less expensive than core drilling. It is appreciated that resistivity measurements cannot be substituted for core drilling when samples are required.

TABLE I.—COMPARISON OF RESISTIVITY METHOD AND CORE DRILLING.

Method	Approximate Operation Cost per Day	Depth Exploration			Location of Edge of Rock	
		"Holes" per Day[a]	Footage per 8-hr. Shift	Approximate Cost per foot	Time per Determination, days	Approximate Cost per Determination
Resistivity	$115	4	200	$0.60	1/4	$30
Core drilling	75	1	50	1.50	3	225

[a] Hole units are assumed to be 50 ft. in depth.

In comparing drilling and electrical resistivity for determining the boundaries of rock of this nature, the resistivity method has a decided advantage. One line of fixed spacing traverse observations is sufficient to locate the edge of the rock deposit and can be completed in about two hours, while it would take a minimum of three drill holes to achieve the same purpose. The latter would require about three days, which makes the resistivity method approximately twelve times faster than drilling. These data are summarized in Table I.

Aiken "Quartzite" Quarry (Nebr.):

The Aiken quarry is located near the right abutment of the Medicine Creek Dam, currently under construction near Cambridge, Nebr.

From an examination of the outcrop which was variable and rather thin, the quarry did not present a very promising appearance. It also had an overburden, which was more than 50 ft. thick in some places. But, because of the possibility of using part of the overburden in the dam embankment, a core-drilling program supplemented by the resistivity method was undertaken to map the rock deposit.

Twenty-two resistivity depth measurements and over 1500 ft. of fixed depth traverse were run in a period of 9 days in May, 1948. The geophysical work was planned to take advantage of the information from and to supplement the drilling. Preliminary resistivity measurements were made at the outcrops of the silicified rock and at strategically located drill holes to establish control for the interpretation of the geophysical measurements. It developed later that where the loess overburden exceeded 30 ft. the effectiveness of the method was restricted, partly because of the thinness of the rock layer. The work had therefore to be planned to take advantage of drainage channels and other locations where the overburden did not exceed 30 ft. In spite of this restriction, the approximate thickness and areal extent of the rock deposit was successfully determined.

In one instance during the progress of the exploration, a fixed depth resistivity traverse was run up a small valley, a distance of over 750 ft. from an outcrop. The high resistivity indicative of the silicified rock was found along nearly the entire length of the traverse. The thickness of the layer was then checked by several resistivity depth measurements. As a result, five drill holes were located along the traverse line. Drilling proved that a layer of silicified rock 4 to 5 ft. in thickness was present and, in this manner, approximately 30,000 cu. yd. of potentical riprap material were located in this area. In this instance, the electrical resistivity method definitely pointed the way for the drilling.

Herndon and Ludell "Quartzite" Quarries (Kans.):

These silicified sandstone deposits were investigated in May and June, 1948 as possible sources of riprap for use in the construction of Bonny Dam at Hale, Colo., a few miles west of St. Francis, Kans. The deposits are located near the town of Herndon in northwestern Kansas. Here again, the sandstone exhibited variations in hardness, silicification, and fineness of grain. Some of the rock layers were separated by zones of uncemented sand. At some points, as little as 25 ft. apart, cores of silicified sandstone were obtained which ranged from 6 in. to 9 ft. in length.

During 14 days of field work, 29 resistivity depth curves, plus over 2200 ft. of fixed-depth traverse were completed. Drill holes and jackhammer holes were used as controls to supplement the resistivity information. This work permitted an estimate of the quantity of rock and the thickness of overburden. In this case, there was no project very close to the deposit, and the stripping would have had to be wasted.

The Ludell quarry, which lies approximately ten miles west of the Herndon deposit, is much smaller in size but somewhat more consistent in quality. In three days' work, nine resistivity depth curves and approximately 825 ft. of traverse were completed, which, in addition to previous test pitting, permitted the available yardage to be estimated.

Miscellaneous:

Several other similar deposits, located in Kansas and Colorado, were successfully explored by this method. A total of approximately 1,000,000 cu. yd. has been mapped so far during one construction season in seven different explorations.

APPLICATIONS OF THE RESISTIVITY METHOD TO THE INVESTIGATION OF GRAVEL DEPOSITS

The use of the resistivity method for outlining sand and gravel deposits was presented by S. W. Wilcox in 1944 (2). The use of the method for this purpose is based on the fact that the resistivity of sand and gravel ranges ordinarily from 1000 to 20,000 ohm ft. while that of the surrounding soil, clay, or shale ranges from 50 to 200 ohm ft. This provides the contrast in physical properties which permits the successful use of the resistivity method. Expensive test pitting is reduced to a minimum and a good estimate of the quantity, depth, and overburden can be effectively made.

In the fall of 1948, successful use of the method, as described by Wilcox, was made in the exploration of a gravel deposit near Ft. Morgan, Colo., for material needed for the proposed Narrows Dam. A geologic section of the gravel exposure is shown in Fig. 9. The first step was to obtain the resistivity of the gravel and other beds at the outcrop, using resistivity measurements with an electrode spacing of from 4 to 6 ft. The sharp contrast in the resistivity of the different layers in the exposed section is indicated in this figure. Control drill

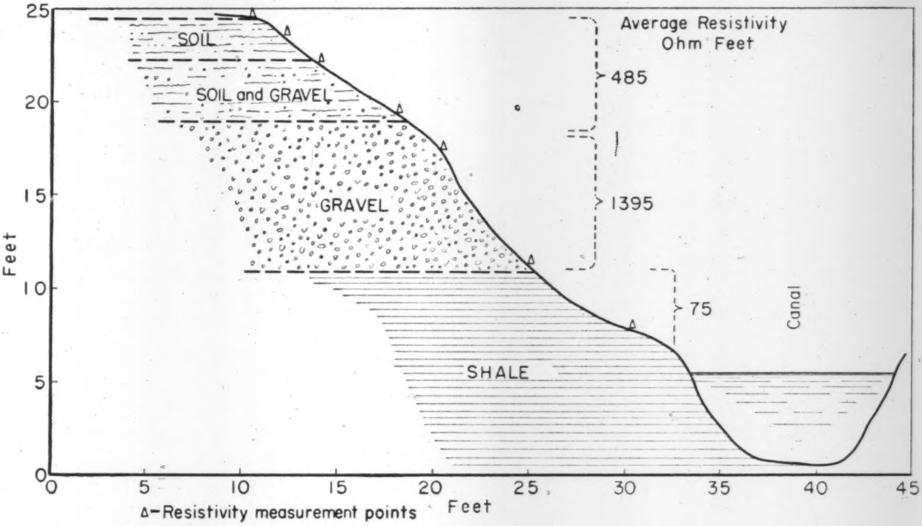
Average Resistivity
Ohm Feet
485
1395
75
SOIL
SOIL and GRAVEL
GRAVEL
SHALE
Canal
Feet
0
5
10
15
20
25
30
35
40
45
Δ-Resistivity measurement points

Fig. 9.—Geologic Section and Resistivity Measurements at Gravel Outcrop Near Ft. Morgan, Colo.

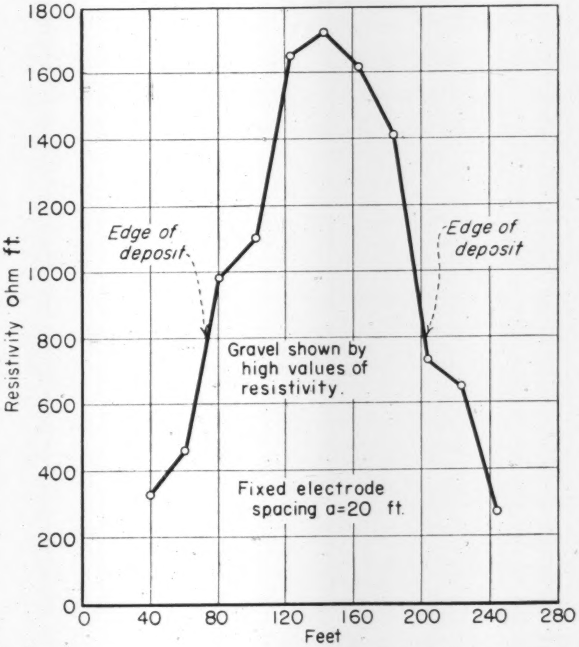

Fig. 10.—Fixed-Depth Resistivity Traverse at Gravel Deposit Near Ft. Morgan, Colo.

holes were used in a manner similar to that in the previously described rock deposits. Resistivity depth measurements and fixed-depth resistivity traverse lines were made to locate the depth and thickness of the deposit at various points and to outline the edges of the gravel body. An example of such a traverse is shown in Fig. 10. The cost of the work was comparable to that in the rock deposits.

SUMMARY AND CONCLUSIONS

It is recognized that the resistivity method cannot replace physical samples for the determination of the character of the materials. However, the fact that, by their nature, the observations give "summation" data rather than "point" data obtained by drilling, makes the information more valuable for an over-all appraisal of a given deposit. Based on Bureau of Reclamation experience, the following conclusions may be reached:

1. The presence or absence of silicified rock or gravel under conditions similar to those presented in this paper can be established by resistivity depth measurements. The approximate depth and thickness of such layers can be estimated from an inspection of the resistivity depth curve provided control is available either through exposures or through drill holes or test pits. The soundness of the interpretations depends on such controls.

2. The boundaries of such rock or gravel deposits can be established by the use of constant depth resistivity traverses.

3. The resistivity method, as applied in the examples given, effected definite savings in time and money in the exploration of these materials.

4. The results demonstrate that the resistivity measurements can, in most cases, lead the way in explorations and materially reduce drilling by permitting the location of drill holes most strategically.

5. The resistivity method, because of its simplicity, does not require highly trained field operators to assist the geophysicist.

6. The work reported in this paper shows conclusively that the resistivity method, supplemented by a limited amount of drilling or test pitting, furnishes adequate data to permit mapping such materials deposits, as a basis for a realistic estimate of the quantities (except for certain very special cases) in a shorter time and at less cost than other currently used methods of exploration.

APPENDIX

THE RESISTIVITY METHOD

The resistivity method of electrical prospecting was developed by Messrs. O. H. Gish and W. S. Rooney (3) for the purpose of making measurements of the resistivity of the ground. The procedure was worked out during investigations of natural earth electrical currents in about 1924. In studying these earth currents, it was necessary to determine quantitatively the resistivity of the subsoil at given points. The equipment and field procedure developed for measuring ground resistivity were later found to be adaptable, without modification, to the solution of a number of field exploration problems.

The method is essentially a field application of Ohm's law, which states that when a direct current flows in an electrical circuit, the resistance, R, in ohms equals the voltage, V, divided by the current, I, in amperes. In equation form, Ohm's law is:

$$R = V/I \qquad (1)$$

To apply this to a body of earth materials, only five items of equipment are required.

Item	Remarks
1. Four ground contact electrodes	Two types, current and potential
2. A source of direct current	45-v. B-batteries
3. A milliammeter	To measure current (*I*)
4. A potentiometer	To measure voltage (*V*)
5. Four reels containing suitable lengths of lead wire	Lightweight single conductor rubber covered flexible copper wire

The Resistivity of Materials:

The electrical resistivity of materials is a physical property that varies over a wide range. For example, the resistivity of wet clay may be 16 ohm ft. and that of granite 19,000 ohm ft. The resistivity of a given material depends on a number of factors such as (1) the resistivity of the constituent grains or particles, (2) the porosity, (3) the resistivity of the fluids, if any, filling the pores. This last factor is of particular importance and may in itself determine if a given material has a high or a low resistivity. The resistivity of even slightly saline waters is very low, and it is inversely proportional to the salt content. Therefore, the electrical character of the fluids and the degree of saturation of a material is quite critical in determining its resistivity.

TABLE II.—RESISTIVITY OF TYPICAL MATERIALS (4).

Material	Resistivity, ohm-feet	Remarks
Granite, syenite, marble, quartzite	100 000 to 30 000 000	Hard, dense rocks
Sand	3 200 to 32 000	Dry
Sands and gravels	3 000 to 15 000	Wet
Sandstone, limestone	300 to 13 000	Sedimentary rocks
Shale	300 to 13 000	
Groundwater	30 to 3 000	
Clay	650 to 1 000	
Wet clay	300 to 600	
Soil	15 to 30	Moist, loamy

Because of these factors, tables of resistivities of various materials are of limited usefulness. It is ordinarily not possible to determine from such a table if there will be sufficient resistivity contrast to permit the use of the method for outlining a particular deposit. It is therefore necessary to make field tests or laboratory measurements on carefully taken samples.

Table II gives broad ranges of values of resistivity of different classes of materials which are based on a large number of measurements.

Procedure:

To make a resistivity measurement in the field, the equipment is arranged as shown in Fig. 4. The four electrodes are first set in the ground in a straight line at equal distances from each other. The B-batteries and the milliammeter are then connected in series with the outer pair of electrodes (or current electrodes) using the wires on two of the reels. The potentiometer is also connected between the inner pair of electrodes (potential electrodes) by means of the wires on the other two reels.

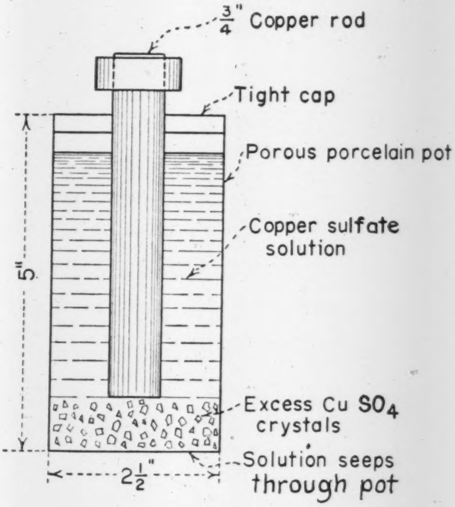
$\frac{3}{4}$" Copper rod
Tight cap
Porous porcelain pot
Copper sulfate solution
5"
Excess Cu SO_4 crystals
Solution seeps through pot
$2\frac{1}{2}$"

FIG. 11.—Porous-Pot Electrode.

The current, *I*, that flows between the current electrodes is measured on the milliammeter and, at the same time, the voltage, *V*, between the potential electrodes is measured on the potentiometer. The equal electrode spacing, *a*, is measured in feet.

The resistivity ρ of the soil or rock included in the measurement is $2\pi a$ times the ratio of the voltage and current, V/I:

$$\rho = 2\pi a\, V/I \qquad (2)$$

The quantity $2\pi a$ relates to the volume of the material and the V/I ratio is resistance as given in Eq. 1.

In the equipment adopted by the Bureau of Reclamation, two different types of electrodes are used. The outer pair, or current electrodes, are metal pins (about one-half inch in diameter by 12 to 18 in. long). These are driven into the ground to a depth of a few inches to secure good electrical contacts. The inner pair, or potential electrodes, are of special design. As shown in Fig. 11, these consist of small porous porcelain pots (approximately two and one-half inches in diameter and five inches high) provided with a tight cap and filled with a (saturated) solution of copper sulfate. A copper rod (about three-quarters of an inch in diameter and approximately five inches long) is immersed in the copper sulfate solution, and the hole in the cap through which the rod passes is sealed. Such porous-pot electrodes make good electrical contacts with the ground because some of the electrolyte seeps into the soil. An electrode of this type is known as a depolarizing or nonpolarizing electrode. Battery jars which are easily obtainable, in different sizes, are suitable for making such electrodes.

The use of porous-pot electrodes in the potential circuit has proven most satisfactory and eliminates the necessity for a double commutator, which is provided in most sets of resistivity equipment that are available commercially. This simplifies the apparatus and reduces its cost. The purpose of the commutator is to permit the use of four metal electrodes by eliminating the current produced by electrolytic action at the metal potential electrodes where they are in contact with the ground. This is accomplished by the commutator which reverses the direction of the current and the voltage at the rate of about 15 to 20 cycles per second. In either type of equipment, the voltage cannot be measured by an ordinary voltmeter but requires a potentiometer which has the advantage that it does not draw any current when a reading is made.

Field Techniques:

The two techniques most commonly used in the application of the resistivity method are (*a*) resistivity depth measurements and (*b*) fixed-depth resistivity traverses.

Resistivity depth measurements.—A resistivity depth measurement is made by taking a series of readings of current and corresponding voltage at various electrode separations to determine the changes in resistivity with depth at a selected location. As the electrode separation is increased, the current penetrates to a greater depth and a greater volume of material is therefore included in the measurement. An example of the observations for such a resistivity depth determination is given in Table III.

The values of resistivity calculated from the above measurements show that as the electrode spacing, a, was increased, the resistivity became greater. In this example, the fact that the deeper layers of material had a higher resistivity than the shallower ones is apparent, as the value of the total resistivity increased with depth or when measured at the larger electrode intervals. This might represent the case of low-resistivity moist surface layer underlain by high-resistivity dry material.

The reverse might equally well have been true. A dry, or highly resistant, layer at the surface with a moist low-resistivity zone beneath would show decreasing values of resistivity at greater electrode spacings.

TABLE III.—RESISTIVITY FIELD DATA.

Electrode Separation, a, ft.	Voltage, v, mv.	Current, I, ma.	Resistivity, ρ, ohm-feet
3	170	5	636
5	290	10	912
10	228	11	1310
20	150	10	1880

In practice, the initial readings of current and voltage are made with the current flowing in one direction. Check readings are taken with the current reversed and the average resistivity is used. This permits the effect of any ground electrical current to be averaged out.

As applied in the field, readings are taken first at small electrode spacings of say one to two feet. With the center point of the line of electrodes remaining stationary, the electrode separation is increased progressively by 5-, 10-, or 25-ft. increments until the desired electrode interval (or depth) is reached.

The resistivity values are calculated as the work progresses. Slide rule accuracy is sufficient. From a set of such measurements, the resulting resistivity values are plotted in the form of a resistivity depth curve (or *R-D* curve). As indicated in Fig. 6, the electrode separation, a, appears on the vertical axis and the resistivity, ρ, on the horizontal axis of the graph. As shown in this example, which was taken near a drill hole, a resistivity depth curve is (effectively) a form of electrical drilling since it expresses the variation in resistivity, and therefore in materials or conditions, with depth.

The analogy to electrical drilling is useful but not strictly accurate. The resistivity measurements are, of necessity, made on the ground surface. Hence, a resistivity depth curve does not show the resistivity of individual layers below the surface, but rather the changes of resistivity with depth as a progressively larger volume of material is included in the measurement.

This concept is important, and its significance is illustrated by the field data in Table III. For the volume of material measured at an electrode separation of 3 ft., the resistivity is 636 ohm ft. At an electrode separation of 5 ft. (which took into the measurement a greater volume of material), the resistivity was 912 ohm ft. The measurement at the 5-ft., interval included all the material involved in the first reading (at the 3-ft. spacing) plus a certain amount of new material. The resistivity of the added material was therefore of a higher value than the material measured at the 3-ft. spacing, since it caused an increase in the measured value at the 5-ft. spacing. In each reading at an increased electrode separation, all the material in the previous reading is included plus some new material. Figure 6 brings out this point clearly.

Fixed-depth resistivity traverses.—In this technique, a certain electrode separation is selected and readings are made (maintaining this spacing) progressively along a traverse line across the area to be studied. An example of such a fixed spacing traverse is shown in Fig. 8, where the constant electrode interval was 15 ft.

The resistivity values, measured, are plotted on the vertical axis of the graph and the location of the points of observation on the horizontal axis. Such fixed-depth traverses are analogous to "electrical trenching" in the same manner that resistivity depth measurements can be thought of as a form of "electrical drilling".

Figure 8 shows that, when highly resistant siliceous rock is included in the measurement, the values of resistivity (at the 15-ft. spacing) are 400 ohm ft. or more. Where no such rock is present, as in the vicinity of drill hole No. 2, the resistivity is about 100 ohm ft. or less. As noted, all measurements were made at the constant 15-ft. electrode interval.

The silicified rock in this case was being investigated as a possible source of riprap. The absence of high-resistivity rock, as indicated by the log of drill hole No. 2, is confirmed by the low values of resistivity shown by the resistivity depth curve at that locality, Fig. 7.

These two techniques make the resistivity method very flexible. As most expedient in a given field problem, it is possible to measure the variation of resistivity with depth or to study the changes from point to point in the resistivity of earth materials at a fixed depth.

Types of Resistivity Depth Curves:

Resistivity depth curves ordinarily fall into two categories namely, two-layer curves and three-layer curves. A single layer curve, although possible, would have no meaning, as the resulting graph would be a straight line parallel to the depth axis.

Two-layer resistivity curves are of two possible kinds only. In one, the top layer is of higher resistivity than the lower layer, as in Fig. 7. The other type of two-layer curve is one in which the resistivity of the upper layer is less than that of the lower layer. It is of note that the resistivity measured at the larger electrode spacings, in a two-layer curve approaches the value of the resistivity of the lower layer, as an asymptote.

Three-layer resistivity depth curves are also of two kinds only. In one variety, the top layer is of higher resistivity than the second layer and the bottom, or third, layer is also of higher resistivity than the second layer. The depth sequence in resistivity values of the layers would be high-low-high.

The second type of three-layer resistivity depth curve is shown in Fig. 6. Here, a low-resistivity surface layer is underlain by a high-resistivity second layer, while the third layer is of a lower resistivity than the second. The depth sequence of the resistivity of the layers is low-high-low.

In some cases, four-layer resistivity depth curves are obtained. These are not, ordinarily, useful in shallow work of the kind considered in this paper.

Interpretation of Resistivity Measurements:

Resistivity depth curves can be interpreted by inspection (qualitatively) and valid conclusions reached that are sufficient for many types of work. For example, the curve given in Fig. 6 shows conclusively that a high-resistance material is present below the surface at the locality where the measurements were made. In like manner, the low resistivity values found in the curve given in Fig. 7 demonstrates that no high-resistivity material is present. Such qualitative interpretations can be made only when controls provided by drill holes or outcrops are available. Obviously, based on such controls, qualitative interpretations can be extended to other parts of a given deposit. The interpretation of fixed depth resistivity traverses is always qualitative.

The authors do not consider that a detailed discussion on the quantitative interpretation of resistivity depth curves would be in order in this paper, inasmuch as only qualitative interpretations were required in the work from which the above examples were taken. However, it is worthwhile to note that two- and three-layer resistivity depth curves can be interpreted quantitatively and layer depths and layer resistivities can be determined with satisfactory accuracy. This is done by analyzing the curves and the data obtained. There are several procedures for this purpose, one of which, the Tagg (**5**) method, is quite successful. The Tagg method is based on a two-layer curve but can be extended to the three-layer case. It requires the solution of a number of simultaneous equations. The application of the Tagg method is time consuming as it is, in effect, a "cut and try" process. It requires from 4 to 6 hr. to make one Tagg interpretation. Other methods which have been used successfully for quantitative interpretation are those set forth by Roman (**6**) and Wetzel and McMurry (**7**)

A simple empirical method for quantitative determination of layer depths from resistivity curves has been devised by R. W. Moore (**8**). It is unfortunately, not suitable for all cases; but where it does apply, it is rapid and reliable. Layer depth interpretations can be made graphically by Moore's method in the field. Additional information on the resistivity method, its theory and applications may be found in books by J. J. Jakosky (**9**) and C. A. Heiland (**10**).

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DISCUSSION

Mr. Roland F. Beers[1] (*presented in written form*).—The concept, "resistivity," is not adequately defined in the paper, and the factors contributing to values of observed relative resistivity of a rock formation are not completely shown. Neither are relationships developed so as to express the significance of these factors individually or in conjunction.

Failures of the method have been observed in practice. Their causes cannot be understood without a more fundamental exposition than is given here.

The utility of resistivity surveys in the selected applications would be enhanced by reference to geologic mapping of the areas surveyed. Only briefly does the paper deal with formation names. The lithology of most geologic formations is well known to competent geologists. In this field of data will be found much valuable aid for those in search of construction materials. Furthermore, correlation of geologic and resistivity data usually develops enhanced significance of both.

Mr. H. R. Joesting[2] (*presented in written form*).—The paper convincingly demonstrates the value of resistivity surveys, used with drilling, in searching for construction materials which are covered by overburden. The close coordination of geophysical measurements and drilling permitted outlining the boundaries of rock and gravel deposits with a high degree of certainty and precision, and at the same time permitted elimination of a large proportion of the drilling that would otherwise have been necessary.

The discussion of resistivity methods in the Appendix should be of special interest to highway engineers and others faced with the problem of finding construction materials in areas where they are covered by overburden.

More information might have been obtained with little additional effort if the authors had used the so-called partitioning configuration of electrodes instead of the Wenner configuration, which is shown diagrammatically in their Fig. 4. The partitioning configuration merely uses an additional potential electrode, placed midway between the two potential electrodes of the Wenner configuration. This permits measurement and comparison of apparent resistivities of two volumes of material at each setup, that is, between the central electrode and each of the outer potential electrodes, instead of a single volume between the two outer potential electrodes. A comparison of the two resistivities will often give more detailed information on vertical and horizontal changes in the underlying material than can be obtained from a single value.

The statements concerning the speed of resistivity surveys are overconservative. In areas of low to moderate relief and with a trained crew of field assistants, it is normally possible to run at least 20 sets of resistivity-depth measurements to 50 ft. each day, or about 2000 ft. of horizontal profiles. The rate will vary, of course, depending on the time required to move from one setup to the next and on the amount of detail required. At any rate, on a routine resistivity survey lasting long enough to permit training moderately agile field assistants, the unit cost of resistivity work is considerably lower than stated.

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[1] Professor of Geophysics, Head of Dept. of Fuel Resources, Rensselaer Polytechnic Institute, Troy, N. Y.

[2] U. S. Geological Survey, Washington, D. C.

MR. L. E. GREGG.[3]—I feel that there are applications of this resistivity method which can be made now and are not being made merely because they have not been brought to the attention of the soils engineers and others.

For example, in Kentucky we have about a fourth of the entire area of the state—confined entirely to the eastern part—in which there are vast expanses or deposits of sandstone that are variable in all directions and all manners. For many years attempts have been made to use this material. We know a good deal about it in certain localities and have used it to a limited extent in highway construction.

However, because it is variable, and because of the fact that there has never been any commercial development, its use is so limited that we actually still know little about it. And, when we try to make an application of the material, it means intensive study of each location under consideration.

Now, with a method such as this, I feel that we ought to be able to learn more in less time about this stone and perhaps realize many benefits that we haven't had heretofore.

The same is true of unconsolidated deposits of fill material in some of our river valleys where we have to carry foundations for large bridges to rock. Sub-surface explorations would be expedited by the application of a method such as this.

Another thing that occurred to me was the possible application of this to soils in regions where we have found through experience that problems of a certain nature exist. I am thinking particularly of one location in central Kentucky where we have soils in general derived from the weathering of limestone. They are fine grained and of a peculiar structure as you probably know.

Within these soils, in certain localities, are sand pockets that apparently were formed by the disintegration of sandstone at the time of the formation of the fine-grained soils that predominate. It is difficult to determine just where these occur, and we have found by experience in construction that many of our slopes in deeper cuts are unstable, even when we lay them back more than usual. Now, that is something that we haven't been able to determine at the time of location surveys on the site.

The location is set, right of way is bought, and the soil survey preliminary to design is made before we are aware of these subsurface conditions. Sometimes they may not be apparent until after construction begins.

We may find these conditions and then realize that we have need for more right of way in order to lay those slopes back. We could possibly make use of this method for a rapid and inexpensive survey of the area at the time of location so that those conditions could be taken into account at the stage in which we are buying the right of way, instead of having to go back after the right of way has been purchased and attempt to compensate for this lack of information.

MR. E. A. ABDUN-NUR (*author*).—With regard to the comments by Mr. Beers, we intentionally stayed away from the highly technical treatment of the subject, because we are aiming to make it of practical value to the engineer who is not a geophysical specialist. Theoretical fundamentals would, we felt, be out of place in this type of paper.

With regard to the use of the unit

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[3] Associate Director of Research, Kentucky Department of Highways, Lexington, Ky.

"ohm centimeters" instead of "ohm feet," we find the latter more practical with our men in the field who survey in the foot units.

As suggested by Mr. Beers, we do have geologists to help us with the investigations. In fact our materials investigation group is part of the geology section. But the work reported in this paper is not a case of trying to find materials. The examples we have given are instances where we knew the materials existed at certaion locations. It was a question of mapping, estimating, and evaluating them for construction purposes in order to be able to inform the contractor of the variations, extent, quantities and other items which influence his bid and methods of operation. We actually make specification maps of the materials deposits, showing the configuration of the rock ledges, thickness, and variations. That is the reason for the need of the detailed investigations. Geological treatment would be too general for that purpose.

Mr. Joesting's comments and the paper presented by Mr. Shepard at the 1949 Annual Meeting[4] indicate that this type of work can be pursued at a more rapid rate than we have quoted. The figures given in this paper are however all inclusive. They include the time needed for interpretations and the estimating of the quantities.

MR. BRYANT MATHER.[5]—Although I am an employee of the Corps of Engineers, the following discussion is presented by me as an individual and has no official sanction. In the previous discussions mention has been made of the desirability of having information of the sort presented in the paper made available to provide examples of the type of work that can be done and to serve as an explanation of the methods used.

I should like to invite your attention to a publication that is already available, having been recently published by the Waterways Experiment Station. The publication is *Bulletin No. 33* entitled "Electical Resistivity Exploration" written by Mr. William R. Perret, Chief, Physical Measurements Section, Soils Division.[6] The first part of the *Bulletin* is devoted to theoretical and general considerations and the remainder to a description and discussion of the application of an electrical resistivity method to a particular exploration problem. The problem in this case may be more closely related to the one concerning soils in Kentucky previously mentioned than to those discussed in the paper, since it deals exclusively with soils.

The problem discussed in the *Bulletin* is that of determining the thickness of recent alluvium in the Mississippi Valley along the main line levee system in the Vicksburg, Miss., District as part of a study of the seepage under levees. The project involved nearly 200 determinations of depths up to 150 ft. along a line approximately 40 miles in length. The work was accomplished in 1942–43 at a cost of approximately 45 per cent of the cost for an equivalent boring. The average time required for a depth determination by resistivity methods was $\frac{2}{3}$ days as compared with an estimated 4 days for completion of a boring to the base of the alluvium.

MR. C. M. WAKEMAN.[7]—I should like to ask the author what limitations, if any, are there with respect to the amount of moisture in the soil, for example, a dry soil compared with a saturated soil.

MR. ABDUN-NUR.—The resistivity is

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[4] E. R. Shepard, "Subsurface Explorations by Geophysical Methods," *Proceedings*, Am. Soc. Testing Mats., Vol. 49, p. 993 (1949).

[5] Concrete Research Engineer, Concrete Research Div., Waterways Experiment Station, Corps of Engineers, Jackson, Miss.

[6] Available from the Waterways Experiment Station, Vicksburg, Miss., for $1.00 per copy.

[7] Testing Engineer, Los Angeles Harbor Department Testing Laboratory, Wilmington, Calif.

lower in a saturated soil. In the table presented in the Appendix, dry sand gives a resistivity value ranging up to as high as 32,000 ohm-feet, whereas wet sand shows 15,000 ohm-feet; the wetter the material, as a rule, the less resistance it will have.

MR. WAKEMAN.—Could the method be applied to submarine exploration?

MESSRS. E. A. ABDUN-NUR AND DART WANTLAND (*authors' closure*).—As pointed out by Mr. L. E. Gregg, there are indeed numerous other specific applications of this method to engineering problems and related fields. Two such excellent examples may be cited. The first is the *Waterways Experiment Station Bulletin* by Mr. Perret, referred to in the discussion submitted by Mr. Bryant Mather. The second example is the determination of the effective change in the density of the material in a waste dump due to rolling as noted in Mr. M. N. Sinacori's discussion of the paper by Mr. Shepard which appears in the A.S.T.M. *Proceedings*, 1949, p. 1014.

The authors appreciate the suggestion made by Mr. Joesting concerning the advantages of the Lee partitioning method. This configuration of electrodes permits obtaining additional information with very little more effort. It is planned to alter the existing resistivity equipment used by the Bureau of Reclamation to permit such measurements.

With regard to the question raised by Mr. C. H. Wakeman, it is felt that the electrical resistivity technique discussed in this paper is not applicable for submarine explorations in relatively deep water. However the method was successfully applied in exploring the bottom of the Harbor of Algiers in shallow water.

The authors wish to express their sincere appreciation for the valuable suggestions made in the various discussions.

EFFECTS OF ELEVATED TEMPERATURES ON STRENGTH OF THERMOSETTING PLASTIC LAMINATES*[1]

BY N. FRIED,[2] R. R. WINANS,[2] AND L. E. SIEFFERT[3]

SYNOPSIS

This paper presented the results of an extensive study of the progressive effects of high-temperature conditioning on the flexural and compressive strengths of a glass-melamine and two different types of phenolic laminate. Tests were conducted at elevated temperatures ranging from 50 C. to 250 C. after conditioning periods of from 1 to 768 hr (32 days). Methods and special equipment for making measurements at high temperatures are described in detail.

Results are discussed and arbitrary criteria set down for determining the maximum operating temperature (M. O. T.) of the materials. Based on these criteria, it was found that the laminates investigated had the following M. O. T. ranges: glass-melamine, 150 to 200 C.; paper-base phenolic, 100 to 150 C.; fabric base phenolic, 100 to 150 C.

An hypothesis is presented which essays to explain the behavior of the materials in terms of three main effects: softening, cure, and degradation; and further evidence is given to support the theoretical analysis.

In recent years the use of thermosetting plastic laminates has been extended increasingly into applications which inherently involve exposure to elevated temperatures. It has thus become necessary to determine the effects of such exposure on the strength of these materials. These effects may be considered from two points of view: Effects on strength as measured at the elevated temperature, and effects on strength after elevated temperature exposure followed by cooling to room temperature.

Several independent investigators have reported (**1,2,3,5,7,8,9,10**)[4] that flexural, compressive, and tensile strength, as well as flexural and tensile modulus of thermosetting materials all decrease after short-time exposure to elevated temperatures. Long-time high-temperature exposure followed by cooling generally also causes decreased strength (**4,6,7,10**) However, it has been found (**6,7,9,10**) that at certain temperatures, depending on the material, heating for a period of time followed by cooling may enhance strength characteristics so that material so "heat treated" may be stronger than "as received" material. Hausmann, Parkinson and Mains (**7**) found that at 90 C. the flexural strength of phenolic laminates fell considerably

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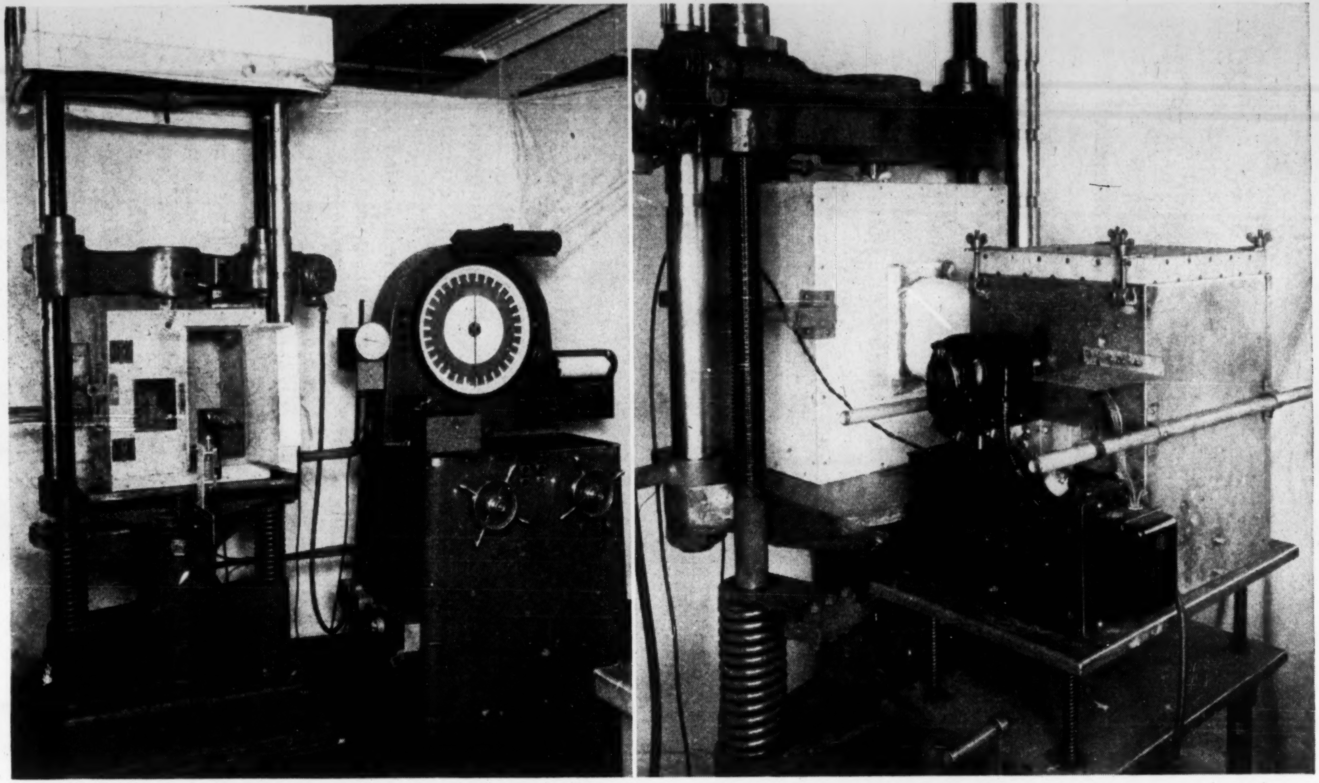
* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] The opinions or assertions contained herein are those of the authors and are not to be construed as reflecting the views of the Department of the Navy or the Naval Service at large.

[2] Plastics Technologist and Materials Engineer, respectively, Material Laboratory, New York Naval Shipyard, Brooklyn, N. Y.

[3] Bureau of Ships, Department of the Navy, Washington, D. C.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1398.



(*a*) Front View. (*b*) Rear View.

FIG. 1.—Apparatus for Determining Flexural and Compressive Strength at Elevated Temperatures.

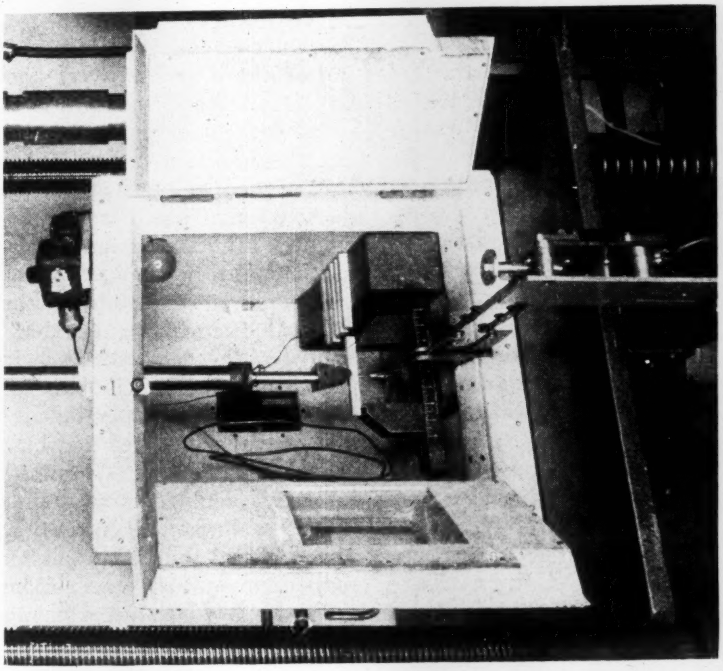


FIG. 2.—Interior View of Test Chamber Set Up for Elevated Temperature Flexural Strength Determination.

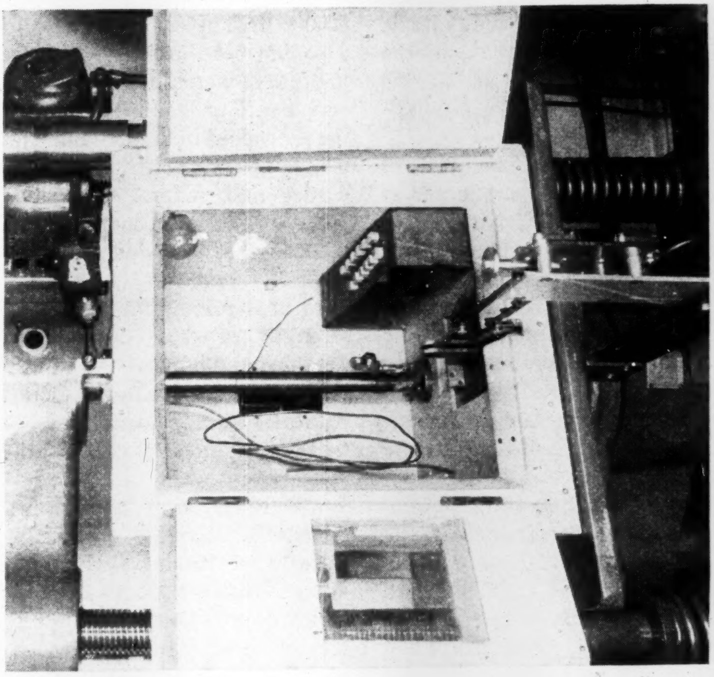


FIG. 3.—Interior View of Test Chamber Set Up for Elevated Temperature Compressive Strength Determination.

from the room temperature value after a short period of time; however, as the time of exposure increased, up to one month, the flexural strength continuously increased.

The present paper covers one phase of a long-range program being conducted at the Material Laboratory, New York Naval Shipyard, to investigate the progressive effects of elevated temperature conditioning on the mechanical and electrical properties of thermosetting laminates as determined both at the temperature and after cooling. This phase includes the results of a comprehensive study of the effects of high-temperature conditioning on the flexural and compressive strengths of several thermosetting laminates, as determined at the elevated temperature. Temperatures covered ranged from 50 C. to 250 C. and conditioning periods from 1 to 768 hr.

MATERIALS

The materials evaluated in these investigations included the following:

Navy Type	Description
FBG	Fabric-base phenolic laminate
PBE	Paper-base phenolic laminate
GMG	Glass-melamine laminate

All materials were obtained from commercial sources and were supplied as sheets approximately 10 sq. ft. in area, with nominal thicknesses of $\frac{1}{2}$ in.

APPARATUS

Except for the specimen supports and loading devices, the same equipment was used for conditioning and testing both flexure and compression specimens. This apparatus is shown in Figs. 1 to 3. The main elements of equipment used in these investigations were as follows:

Testing Machine.—The physical testing machine used for all determinations was a Southwark-Emery Universal Testing Machine of 60,000-lb. capacity, with the following scales: 300, 1200, 6000, 60,000 lb. This machine is equipped with a pacing head used in maintaining loading rate in in. per min., a load maintainer, and a rate of loading device for indicating dial speed in lb. per min. The machine is also provided with a Microformer Recorder and several different types of Microformer Extensometer for automatic recording of load-deflection curves.

Test Chamber.—The test chamber used in the determination of mechanical strength at elevated temperatures was designed and built at the Material Laboratory. It consists of a double-walled, cubical box approximately 2 ft. in dimension on an outer edge. Both the inner and outer walls are made of $\frac{1}{4}$-in. Transite, and their inner surfaces are $2\frac{1}{2}$ in. apart, providing a space into which glass wool insulation is packed. Two doors, which open outward, are mounted in the front of the test chamber, and a window is set into one of these doors for visibility.

An opening is provided in the top of the chamber to accommodate a loading bar which extends into the chamber from the sensitive crosshead of the testing machine. Suitable openings are also provided in the floor of the chamber to permit the insertion of test fixtures. The back of the chamber is provided with two ducts through which heated air may be circulated in the chamber by the air heating unit.

A stainless steel, tubular thermostat unit is located in the test chamber and regulates the temperature within the chamber as described below.

Air Heating Unit.—The air heating unit was also designed and built at the Material Laboratory. It consists of an insulated, double-walled, stainless steel box with blower and duct work for providing air to the test chamber at the proper temperature, under forced draft.

The air is heated in the supply unit by electrical strip heaters and a control grid heater. The 500-w. strip heaters are used as ballast to raise the temperature of the air to a point slightly below that required for testing.

The temperature of the circulating air is raised to the proper point, and maintained at that temperature, by means of a special 800-w. grid heater which is controlled by the thermostat unit in the test chamber. It was found experimentally that this thermostatic control system is capable of maintaining the set temperature to within 1 C. up to 100 C. and to within 2 C. up to 250 C.

Because of the rapid rate of air circulation provided by the blower in the air heating unit, and because of the high thermal capacity of the strip heaters and of the walls of both the test chamber and supply unit, the temperature in the test chamber returns to the established test temperature in approximately 10 to 20 sec. after the doors have been opened and closed to insert a specimen for conditioning or test. As a further check, however, the temperature within the test chamber is measured continuously with a recording potentiometer, and no elevated temperature test is made until this temperature has returned to the required point.

TEST METHODS

Specimens:

Flexure.—Specimens used for determining flexural strength were standard A.S.T.M. flexure specimens 5 by $\frac{1}{2}$ in. by thickness of sheet ($\frac{1}{2}$ in. nominal). The lengthwise direction of the sheet was arbitrarily chosen as that of the longest dimension, and specimens were cut with the 5-in. dimension in this direction.

Compression.—Specimens used for determining compressive strength were approximately cubical, measuring $\frac{1}{2}$ by $\frac{1}{2}$ in. by thickness of sheet. These specimens were cut from bars, $5\frac{1}{2}$ by $\frac{1}{2}$ in. by thickness by sheet, which were prepared in the same manner as were the flexural specimens described above. The "lengthwise" direction was marked by drawing a $5\frac{1}{2}$-in. line along the face.

Randomization:

The sampling procedure used for selecting test specimens was as follows:

(*a*) The required number of specimens were prepared, several specimens being cut from each of as many sheets of a particular material as were supplied.

(*b*) These specimens were placed, in an arbitrary manner, on a large surface, and numbered, temporarily, with a soft crayon.

(*c*) Using a set of tables of random numbers, samples were selected in a randomized sequence.

(*d*) After this selection, temporary numbers were removed, and the specimens were numbered permanently in numerical order. Thus, any group of specimens selected in numerical sequence would represent a random sample of all of the specimens prepared.

General Test Procedure:

In making flexural and compressive strength determinations, the test chamber was set directly on the table of the testing machine. The air heating unit was placed on an adjustable table adjacent to the testing machine, and it supplied heated air to the test chamber through a short, insulated duct. The entire assembly was designed to permit relative movement between the test chamber and air heating unit when the testing machine table rises during a determination.

Compression.—A compression sample under test rested on a flat, hardened steel support which extended through the bottom of the chamber to the testing machine table. Load was applied to the specimen, concentrically, through a 16 by $1\frac{1}{2}$-in. diameter bar which passed through an opening in the top of the chamber and was fixed to the sensitive crosshead of the testing machine. The interior arrangement of the chamber for making a compressive strength determination is shown in Fig. 3.

Flexure.—In the flexural strength determination, the specimen was evaluated as a simple, centrally loaded beam with a 4-in. span. The specimen was supported on a conventional flexure test fixture and load was applied through a loading nose mounted at the lower end of the aforementioned 16-in. loading bar. Both the loading nose and end supports contact the specimen through $\frac{1}{4}$-in. diam. steel pins. The test fixture was supported on an ebony-asbestos block which extended through the bottom of the test chamber and rested directly on the table of the testing machine The interior arrangement of the test chamber for flexural evaluations is shown in Fig. 2.

Load-deflection data were recorded automatically with a Microformer Recorder actuated by a Peters Microformer Extensometer. Deflection was followed by a ball bearing-tipped screw which was threaded into one end of an equi-armed lever, the other end of which was linked to the extensometer through an extension arm.

In a flexure test the ball bearing contacts the under side of the specimen midway between supports, thus following the midpoint deflection under load. In compression tests, the end of the lever is weighted and the deflection device contacts a horizontal arm which is fixed to the loading bar.

The arrangement of the deflection measuring apparatus is shown for flexure in Fig. 2, and for compression in Fig. 3.

Type of Loading.—In these investigations, specimens were loaded in two separate ways, designated as "Face Loaded" and "Edge Loaded," respectively. For face loading, the load was applied normal to a surface, in a direction perpendicular to the plane of the laminae. For edge loading, the load was applied normal to an edge, in a direction parallel to the plane of the laminae. In the case of edge-loaded compression specimens the load was applied in the "lengthwise" direction as indicated by the line on the face of the specimen.

Rate of Loading.—The rate of loading used in both compression and flexural tests was 0.05 in. per min., which speed was set with the machine running idle, in accordance with A.S.T.M. requirements.

Number of Specimens.—Five specimens, randomized as previously described, were evaluated under each set of conditions, and the arithmetic mean of these determinations was taken as the strength value under the particular conditions concerned.

Calculations:

Flexural Strength.—Flexural strength was taken as the maximum fiber stress calculated from the load at the moment of break, in accordance with A.S.T.M. Method D 790 - 45 T.[5]

Compressive Strength.—Compressive strength was calculated by dividing the maximum compressive load supported by the specimen, by the original cross-sectional area of the specimen, the result being expressed in pounds per square inch.

All measurements of specimen dimensions were made immediately after preconditioning, and thus represented "as received" values. Measurements of width and depth were made to 0.001 in. and, for flexure tests, the specimen supports were set at 4 ± $\frac{1}{32}$ in.

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[5] Method of Test for Flexural Properties of Plastics (Tentative). This tentative method, as revised in 1949, appears in the 1949 Book of A.S.T.M. Standards, Part 6, p. 119.

CONDITIONING PROCEDURES

Preconditioning..—All samples, after being cut and randomized, were exposed to the standard laboratory atmosphere (25 C., 50 per cent relative humidity) for a minimum of 168 hr. prior to test or elevated temperature conditioning. This preconditioning period is designated in this paper as "C-168/25/50."

Initial Values.—Initial values of flexural and compressive strength were determined by testing samples which had been preconditioned as described above (C-168/25/50) but which had not been exposed to elevated temperatures.

Elevated Temperature Conditioning:

Short Time.—Samples to be evaluated after 1, 2, or 4 hr. of exposure to elevated temperatures were placed on a sheet metal platform in the test chamber, which was maintained at the proper temperature, and conditioned in the chamber itself.

Long Time.—Samples to be evaluated after periods of more than 4 hr. of exposure to elevated temperatures were placed in a commercial circulating air oven maintained at the proper temperature by thermostatic control. One hour prior to the expiration of the conditioning period, samples were transferred to the test chamber, which was also maintained at the proper temperature, and the conditioning period was completed in the chamber itself. Specimens were transferred from the oven to the test chamber in a specially designed, insulated box, which had been brought up to the proper temperature by means of a strip heater contained therein.

Test Chamber Temperature Control.—Temperature within the test chamber was measured by means of a thermocouple and calibrated potentiometer. The thermostat, which is mounted in the test chamber, and which controls the grid heater in the air heating unit, was set to maintain the required temperature, as measured by the potentiometer. As previously indicated, it was found that, by this procedure, the temperature within the test chamber could be maintained to within 1 per cent of a predetermined value.

EXPERIMENTAL

Face- and edge-loaded flexural and compressive strength determinations were made at elevated temperatures on specimens which had been maintained at the required temperature for varying periods of time. Time periods and temperatures covered were as follows:

Time (hr.) ...	1, 2, 4, 24, 48, 72, 192, 384, 768
Temp. (deg. C.)	50, 100, 150, 200, 250

In addition to this, similar determinations were made on specimens conditioned as described above but cooled to room temperature prior to testing. Results, in general, were similar to those reported by previous investigators and, although mention will be made of these results, no detailed data will be presented in this paper.

RESULTS

Results are given in Tables I and II and shown graphically in Figs. 4 and 5, in which per cent of initial strength retained is plotted against time of conditioning.

In general, specimens evaluated after cooling (recovery) had higher mechanical strength than specimens conditioned for corresponding periods of time but evaluated at the elevated temperatures, the respective curves of strength *versus* time being approximately parallel. Several exceptions were noted in the case of the FBG laminate, particularly for specimens conditioned at the higher temperatures for short periods of time.

The GMG material did not comply

TABLE I.—FLEXURAL STRENGTH OF PLASTIC LAMINATES AT ELEVATED TEMPERATURES.

	Conditioning Time, hr.	Flexural Strength, psi.[a]									
		50 C.		100 C.		150 C.		200 C.		250 C.	
		Face	Edge	Face	Edge	Face	Edge	Face	Edge	Face	Edge
PBE	0[b]	19 500	19 900	....	....	....	....	....	....	....	....
	1	16 800	17 100	11 500	11 100	6 850	6 650	3 600	4 400	4 700	5 250
	2	15 300	15 700	9 850	10 800	7 000	7 100	5 300	5 350	2 350	2 950
	4	14 500	14 800	9 650	10 500	7 150	9 250	7 750	6 850	3 000	3 650
	24	13 600	14 900	10 800 19	11 900 19	11 000	9 600	6 400 22	5 650 22	1 900 20	1 650 20
	48	15 800	14 200	12 500	13 600	9 700 50	10 600 50	5 900 49	4 550 49	1 500	1 450
	72	13 800 96[c]	13 300 96	12 000 96	12 200 96	9 900 68	10 200 68	5 950	4 050	....	....
	192	14 900	14 100	12 200	12 500	10 000	8 000	....	....	....	....
	384	14 800	15 100	12 200	13 200	10 600	8 400	....	....	....	....
	768	15 300	14 500	12 900	12 100	8 600	7 900	....	....	....	....
FBG	0[b]	20 800	20 700	...	...	....	....	....	....	....	....
	1	18 600	18 200	11 000	11 800	8 200	8 650	8 000	8 250	7 300	7 100
	2	18 300	18 500	13 300	12 900	7 550	9 400	8 900	8 550	6 500	6 150
	4	17 700	17 900	13 600	13 900	10 300	10 600	8 600	8 100	3 000	2 350
	24	16 700 18	17 200 18	13 600 18	14 000 18	12 000 21	11 900 21	4 750 19	4 050 19	800 20	900 20
	48	14 100	14 600	11 500	12 100	9 750 42	9 450 42	2 800 49	2 150 49	350	450
	72	13 500 96	14 300 96	12 000 96	11 800 96	10 200 69	10 000 69	2 200	1 700	....	....
	192	13 900	13 900	11 800	12 300	7 600	6 800	....	....	....	....
	384	14 500	14 800	12 300	12 400	6 900	5 900	....	....	....	....
	768	12 800	14 200	11 700	11 900	4 700	3 900	....	....	....	....
GMG	0[b]	42 300	55 100	....	....	....	....	....	....	....	....
	1	39 000	51 300	31 100	43 100	27 900	39 200	26 300	36 500	19 500	27 900
	2	37 800	52 000	32 700	43 400	28 200	40 100	23 700	35 800	18 600	26 900
	4	38 100	51 400	32 500	43 900	28 500	42 100	22 000	32 500	16 800	24 700
	24	40 600 18	50 800 18	32 400 18	44 300 18	27 800 21	39 500 21	20 300 20	29 900 20	16 400 20	22 600 20
	48	41 700	53 900	36 800	49 900	25 400 43	35 400 44	19 000 50	27 000 50	15 600	23 200
	72	40 600 96	53 100 96	35 300 96	48 500 96	24 500 65	35 200 65	18 900	27 700	15 700	22 400
	192	41 400	52 900	36 900	48 100	26 600	36 500	19 300	26 000	10 300	14 500
	384	41 400	53 700	36 600	49 400	26 300	35 400	18 000	25 400	5 500	8 350
	768	39 000	52 500	35 200	48 600	24 300	33 200	18 400	24 900	3 100	3 860

[a] Average of 5 observations.
[b] Initial value—tested at 25 C. after C-168/25/50.
[c] Numbers to right of given value represent actual hours of exposure where different from standard conditioning period. For example "19 000 22" would indicate a flexural strength of 19,000 psi. after 22 hr. of conditioning.

TABLE II.—COMPRESSIVE STRENGTH OF PLASTIC LAMINATES AT ELEVATED TEMPERATURES.

	Conditioning Time, hr.	Compressive Strength, psi.[a]									
		50 C.		100 C.		150 C.		200 C.		250 C.	
		Face	Edge	Face	Edge	Face	Edge	Face	Edge	Face	Edge
PBE	0[b]	39 700	23 200	....	....	....	....	....	....	....	....
	1	35 700	19 800	23 400	12 400	16 200	8 100	19 000	8 650	12 800	6 700
	2	35 100	19 500	24 200	13 000	16 100	9 050	20 500	9 250	14 600	7 100
	4	35 200	19 300	25 000	13 400	19 800	11 200	22 200	9 150	17 700	6 100
	24	35 600	19 500	29 600 23	17 600 23	24 800 22	12 000 18	23 600 23	8 850 23	18 700 17	4 400 17
	48	34 500 45[c]	19 700 45	32 100 46	18 300 46	25 900 46	13 000 42	23 100 47	7 550 47	11 600 47	2 150 47
	72	36 200 95	20 200 95	33 200 92	19 200 92	26 500 71	16 400 71	24 300	7 150	....	....
	192	37 500 163	21 200 163	32 800 166	19 500 166	29 700	18 500	....	....	....	....
	384	36 400	20 400	35 100	21 500	30 100	19 200	....	....	....	....
	768	36 800	21 700	34 600	21 600	29 000	18 000	....	....	....	....
FBG	0[b]	41 200	26 900	....	....	....	....	....	....	....	....
	1	38 100	25 600	30 100	22 600	26 300	20 100	21 500	18 200	17 800	16 300
	2	38 300	24 700	31 500	21 700	28 700	21 500	24 500	20 000	20 700	17 200
	4	37 400	25 200	33 100	22 700	31 200	23 000	24 300	19 600	12 100	13 700
	24	40 000 25	26 900 25	36 500 23	26 000 23	29 600 20	24 000 20	19 400 23	14 900 23	520 18	2 150 18
	48	41 200 46	27 100 46	37 300 44	27 800 44	27 300 45	22 000 45	7 500	9 250	125 46	1 150 46
	72	42 900 95	28 500 95	37 300 92	25 600 92	28 900 70	22 400 70	4 500	6 700	....	....
	192	42 500 163	28 600 163	37 000 166	26 300 166	28 300	22 100	....	....	....	....
	384	40 700	27 300	37 900	26 500	22 400	17 800	....	....	....	....
	768	41 100	27 700	35 400	25 600	15 900	12 000	....	....	....	....
GMG	0[b]	94 900	33 700	....	....	....	....	....	....	....	....
	1	91 900	27 800	75 100	26 200	78 000	23 600	89 900	24 500	85 500	17 900
	2	89 200	29 200	77 600	23 200	90 700	23 900	87 200	23 800	83 900	16 500
	4	92 800	29 400	85 800	27 400	92 500	26 000	88 400	20 100	84 700	15 000
	24	97 100 29	29 700 29	95 200 23	28 400 23	95 100 22	24 400	82 100 23	17 500 23	86 000 17	14 600 17
	48	97 300 50	30 000 50	94 900 45	29 300 45	92 100 46	23 400	82 000	11 600	83 600 47	13 400 47
	72	100 900 95	28 900 95	97 300 93	30 100 93	91 600 71	22 800	80 100	15 700	81 100	14 000
	192	101 700 163	31 600 163	95 800 166	29 700 166	88 900	17 300	82 400	13 800	64 000 195	6 950 195
	384	93 500	30 300	95 800	26 600	90 100	17 400	86 300	14 200	59 200 385	5 200 385
	768	93 600	31 500	93 700	28 900	87 600	17 200	80 800	14 500	45 500	1 900

[a] Average of 5 observations.
Initial value—tested at 25 C. after C-168/25/50.
[c] Numbers to right of given value represent actual hours of exposure where different from standard conditioning period. For example "19 000 22" would indicate a compressive strength of 19,000 psi. after 22 hr. of conditioning.

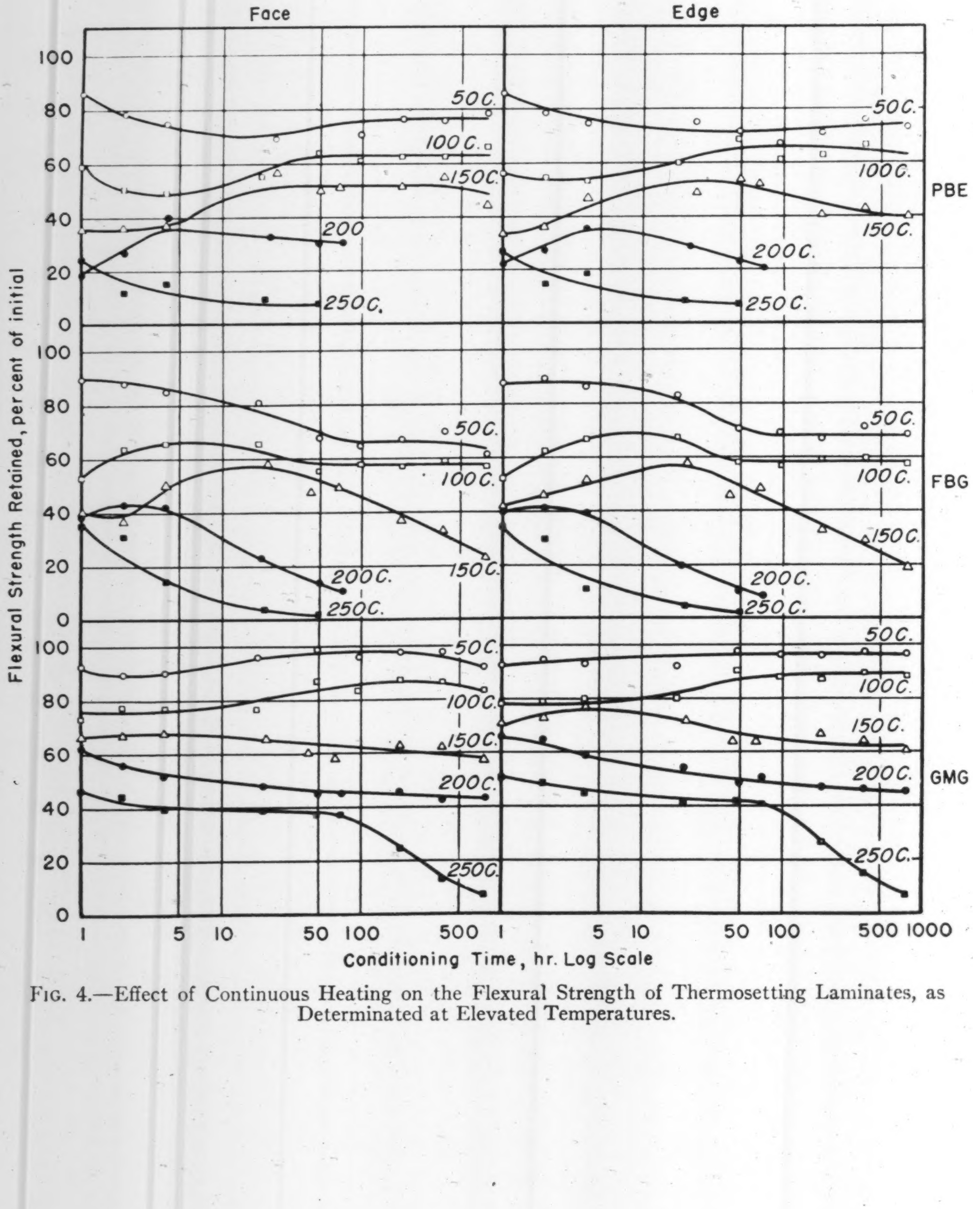

FIG. 4.—Effect of Continuous Heating on the Flexural Strength of Thermosetting Laminates, as Determinated at Elevated Temperatures.

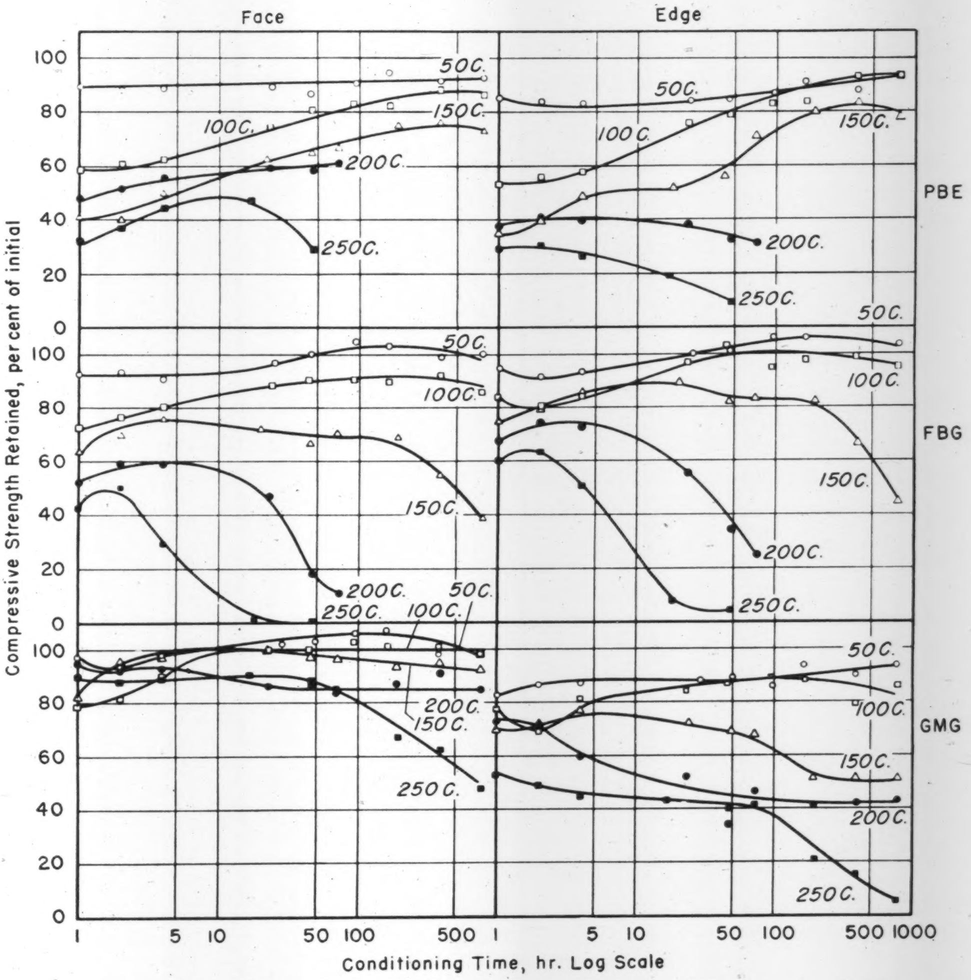

Fig. 5.—Effect of Continuous Heating on the Compressive Strength of Thermosetting Laminates, as Determinated at Elevated Temperatures.

with the above rule too closely, "at temperature" and "recovery" values being close together, with some indication of higher strength when tested at temperature. This behavior was evident, for example, in the GMG edge compression data, particularly for the longer conditioning periods.

Of the three laminates evaluated, the GMG material exhibited the highest initial strength properties. This material also had the greatest comparative physical strength, both at temperature and after recovery, being relatively stable up to 150 C. However, at 250 C. even the GMG material deteriorated rapidly after extended exposure.

Of the two phenolic laminates, FBG and PBE, tests conducted at elevated temperatures showed, in general, that the former had somewhat higher strength characteristics after short-time conditioning, particularly at the lower temperatures. Recovery data showed the PBE to be superior in flexure and, in general, the FBG to be higher in compressive strength, again at the lower temperatures and after short conditioning periods. The particular point of note with respect to these two phenolic laminates, however, is the fact that, for both "at-temperature" tests and "recovery" tests, the FBG material deteriorated at a much greater rate than did the PBE, especially after long-time conditioning at temperatures above 100 C.

MAXIMUM OPERATING TEMPERATURE

A practical purpose of these investigations was to determine the upper limit of temperature which the subject laminates can withstand for an extended period of time, that is, the maximum operating temperature of these materials. This property, which has also been characterized as "heat resistance," has been variously defined in terms of change in appearance or dimension, loss in weight, or change in some strength property. For example, Carswell, Telfair, and Haslanger (6) define the "approximate limiting temperature" of a material on the basis of a permissible 10 per cent reduction in strength after 162 hr. of heating followed by cooling to room temperature prior to test. Hausmann, Parkinson, and Mains (7) used as the criterion for heat resistance the "highest temperature that the material will withstand without visible deterioration or objectionable change in the particular physical property chosen for study." This type of definition has been incorporated into A.S.T.M. Tentative Recommended Practice for Determining Permanent Effect of Heat on Plastics (D 794 – 44 T),[6] which specifies a heating period of 7 days followed by cooling to room temperature prior to evaluation.

As indicated above, the maximum operating temperature (M.O.T.) may be considered, essentially, as the highest temperature at which a material may be continuously maintained, without excessive deterioration. Based on a study of a segment of the data contained in this paper, the following arbitrary criteria for determining M.O.T. were tentatively set up:

Considering measurements made at elevated temperatures, the M.O.T. shall be taken as the maximum temperature (*a*) at which no value of mechanical strength falls below 50 per cent of its initial (C-168/25/50) value after 768 hr. of exposure to the elevated temperature, and (*b*) at which the decrease in any mechanical strength value in the period 192–768 hr. of exposure does not exceed 10 per cent of its initial value.

Further study of the behavior of certain laminates, which dropped sharply in strength after one or two hours of heating but which increased in strength as conditioning time increased, prompted the addition of a further criterion: (*c*) No value of mechanical strength shall, at any time during the conditioning period, fall below 50 per cent of its initial value.

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[6] 1949 Book of A.S.T.M. Standards, Part 6, p. 713.

Based on these criteria, the M.O.T. of the laminates evaluated would be as follows: glass-melamine 150 to 200 C.; phenolics 100 to 150 C.[7]

It is interesting to compare these values with those given by other investigators. Norelli and Gard (8), for example, have stated that phenolic laminates can be used continuously at 100 to 125 C. and intermittently at 150 to 175 C. It has also been stated (7) that high-impact phenolics may be serviceable at temperatures as high as 160 to 170 C. and that cellulose-filled phenolics are good to 130 to 150 C. (3).

Discussion

Examination of the data and curves shows that, in general, at all temperatures, specimens heated for 1 hr. and tested at the elevated temperature, have lower strength than "as-received" samples tested at room temperature, the drop being greater as the temperature increases. Under certain conditions, depending on the material, this may be followed by a period of increasing strength as heating continues. Finally, particularly at the higher temperatures, the strength may fall off again after long-time heating. It is also noted that the modulus parallels this behavior, decreasing initially and increasing or decreasing as the strength varies.

While the detailed mechanisms of the phenomena described above are undoubtedly complex, it is believed that the behavior noted may be explained, qualitatively, in terms of three main effects, in accordance with the following hypotheses:

1. An initial "softening," noted as a strength decrease as well as a decrease in modulus, occurs after a short heating period. This effect is relatively rapid commencing immediately upon heating, and a marked decrease in strength may be noted in several minutes. It is believed that this effect is physical in nature, the resin softening in a manner analogous to the decrease in viscosity of a liquid as the temperature is raised.

2. Additional "cure" may occur in the resin as the material is heated. This is a chemical effect, in which further cross-linking takes place, and manifests itself as an increase in strength and stiffness (modulus). It would be expected that this reaction would be relatively rapid initially but would fall off in rate as available unreacted functional groups were used up.

3. Heating the material may cause scission of bonds, decomposition of resin and filler, and general degradation of material, tending to decrease the strength and modulus. This would be a progressive effect and would be expected to be cumulative as time of heating was extended.

It is recognized that the above hypothetical analysis is considerably simplified and that, in addition, there would be further complicating effects such as might be due to the driving off of moisture, incipient delamination due to expansion, oxidation, etc.

The net behavior noted at a particular point would, of course, depend on the material, temperature and time of exposure. In a given case, the initial softening effect would occur in a short time. If this effect were isolated from all others,

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A strict application of criterion (*c*) would require that the M.O.T. of type PBE material be taken as 50 to 100 C. since the value of face-loaded flexural strength after 4 hr. at 100 C. is 49.5 per cent of the initial value. However, since this is the only respect in which the material fails to meet requirements and since the value is so close to 50 per cent, it is considered that for all practical purposes the given range is justified.

Current investigations of the phenolic laminates at 125 C. show that both materials fail to meet the requirements of criterion (*c*) at this temperature. The M.O.T. for both phenolic laminates may therefore be narrowed down to the range 100 to 125 C.

the strength and modulus would reach a minimum, characteristic of the material and temperature, and would then remain constant, just as, in the absence of extraneous effects, the viscosity of an oil attains a characteristic value dependent on the temperature.

As the temperature rises, reaction rate increases and, under certain conditions, the cure or cross-linking effect may be superimposed on the softening effect, causing a net increase in strength and modulus. Again, in the absence of other factors, the strength would increase with time of heating, rapidly at first but leveling off at a maximum value as the reaction approached completion. It would also be expected that this maximum would be attained more rapidly at higher temperatures.

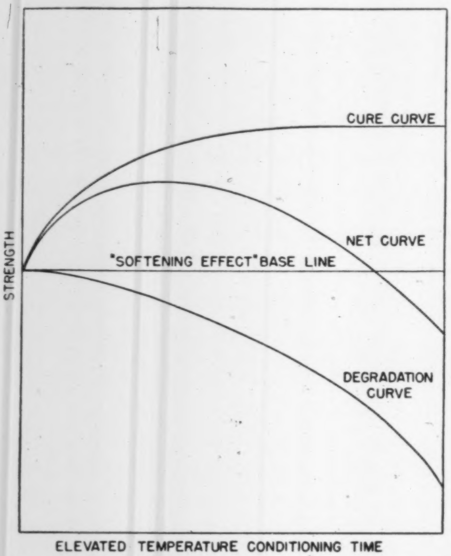

FIG. 6.—Hypothetical "Individual Effect" Strength *versus* Time Curves and Resultant Net Curve.

Finally, the effect of degradation comes in to play, simultaneously with the above, and may cause a net decrease in strength and modulus, depending on conditions. The whole process is undoubtedly dynamic, all of the effects manifesting themselves at the same time, the net test result depending upon the relative preponderance of one or another of the individual effects. Thus at very high temperatures, such as 250 C., the effect of degradation may be so marked as to cause a continuous, rapid decrease in physical properties, masking out effects of cure.

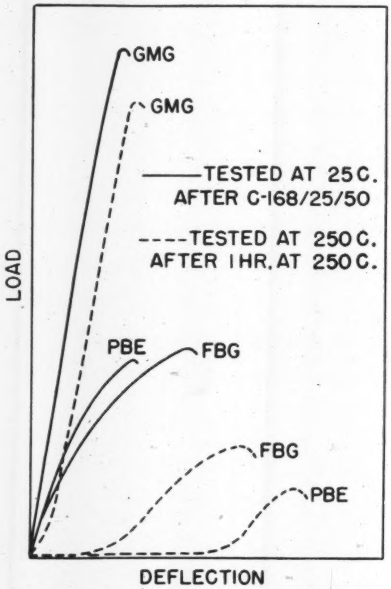

FIG. 7.—Typical Face Compression Load-Deflection Curves for Thermosetting Laminates.

The foregoing hypothesis may be pictured as shown in Fig. 6, which gives a graphic representation of one possible combination of the three effects showing cure and degradation from the "softening effect" base line, and the combined effect which would be noted in testing. Although only roughly qualitative, the shape of the curve is similar to that of curves obtained, under certain conditions, with each of the materials tested. Varying the shape of the separate curves would cause a variation in the net curve approximating behavior actually noted. Thus a steeper "cure" curve would shift the maximum in the net curve to the left, a very slow degradation curve would cause the net curve to increase to a maximum and then level off or fall very slightly after a long time, and a very steep degradation curve would cause a continuous decrease in the net curve, as is actually noted at 250 C.

Further evidence of the softening effect may be seen in the face compression load-deflection curves shown in Fig. 7. "As received" samples, tested at room temperature, give the usual curves which start with a straight line portion, deviate from the straight line at higher loads, and finally break. However, samples tested at 250 C. after one hour of exposure to this temperature give curves of an entirely different shape. In the case of the phenolic laminates there is an extended initial portion of the curve in which a large deflection occurs at low loads; this is followed by an increasing slope which falls off at break. The GMG material behaves in a similar manner, though the initial portion of the curve is less marked. Apparently at 250 C. the resins are relatively soft and compressible; as the resin yields under stress, the filler increasingly takes up the load and the stiffness increases. As a rough check on this behavior hardness measurements were performed on hot specimens and showed an actual physical softening, in some cases after less than 15 min. of heating.

CONCLUDING REMARKS

Recent work at the Material Laboratory has included experience with laminates of other types of thermosetting resins than those included in this report, and it has been found that the aforementioned softening and curing effects occur to a much more marked extent in certain of these materials. It is also interesting to note that preliminary tests conducted after exposure periods of less than one hour show that, under certain conditions, minima in the strength-time curves may occur in as little as 15 to 30 min.

The phenomenon of increasing strength with increasing time of exposure, due apparently to additional cure, would be expected to occur to an extent depending on the degree of initial cure of the material. It is known that in the commercial materials used in these investigations cure was by no means complete. If it were possible to obtain a material which was fully cured, then it would be expected that the strength-time curves would be continuously downward.

Although definite conclusions cannot be drawn at the present stage of the investigation, there is evidence to indicate that the behavior pattern herein discussed may be common, to a greater or lesser extent, to thermosetting materials in general. At present several other types of laminate are under investigation at the Material Laboratory and, if projected plans are realized, this work will be extended to include molded materials. The results of these and similar studies should provide additional data to assist in the analysis of the effects of high temperatures on thermosetting materials.

Acknowledgment:

The authors wish to acknowledge the assistance of J. Kaminetsky who performed the experimental work and compiled much of the data contained in this paper.

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THE EFFECT OF TEMPERATURE ON THE CREEP AND RECOVERY OF A MELAMINE-GLASS FABRIC LAMINATE*

BY W. J. WORLEY[1] AND W. N. FINDLEY[1]

SYNOPSIS

Results of creep tests of a melamine-glass fabric laminate at temperatures of 40, 77, and 130 F. are reported. The relative humidity of the air surrounding the specimens was maintained at about 50 per cent by improved apparatus described. Creep was measured from specimens tested at several different stresses for each of the three temperatures. Dimensional changes under no stress and changes in weight of the material were also recorded.

Correlation of the data with the activation energy theory of creep is examined by means of a new method of analysis. It was observed that the creep resistance of this material was much greater than that of the canvas laminate and rayon laminate previously reported (**1**)[2] and its temperature sensitivity was much less than that of the canvas laminate.

The problem of obtaining accurate creep data on plastics is discussed.

During the interval from the time our first tests were conducted at different temperatures (**1**)[2] in 1945 to the present tests the authors attempted to improve the testing apparatus and technique so that more precise creep test data on plastics could be determined for analysis and comparison with theories of creep.

The influencing factor which had proven most difficult to control in the previous tests (**1**) was relative humidity. Accordingly the laminate chosen for the present tests was selected as having less sensitivity to humidity changes than the laminates previously tested. This laminate was also chosen because of the current interest in its high strength and temperature resistance. Improvements which had been made in the means for controlling relative humidity were used in the current test procedure.

In order to permit more complete comparison of the properties of this laminate with other laminates the stress-strain curves in tension and compression, fatigue strength, and hardness are reported.

Material Tested and Specimens:

The material tested was a glass-fabric laminate molded with a melamine-formaldehyde resin at a pressure of 1250 psi. and a temperature of 284 F. The preparation and composition of the laminate are summarized in Table I.

Dimensions of static tension and compression specimens as well as creep specimens are shown in Fig. 1. All specimens were cut from the sheet with their longitudinal axes parallel to one another and parallel to the direction of greatest strength of the sheet. The creep specimens were cut from the original sheet in such a manner that the flat side of the specimens were perpendicular to the plane of the sheet.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1412.

Control specimens used to determine the magnitude of shrinkage during the test were identical with the creep specimens. The control specimens for determining the change in weight of the material during the test were the same as the creep specimens except that the width throughout corresponded to that of the reduced section of the creep specimens.

All specimens were placed in a controlled-atmosphere room maintained at 77 ± 1 F. and 50 ± 2 per cent relative humidity for at least two weeks after machining before testing was started. (All room-temperature tests were conducted here.)

TABLE I.—PREPARATION AND COMPOSITION OF GLASS FABRIC LAMINATED WITH MELAMINE RESIN.

Designation	Textolite No. 11508 Br.
Laminator	General Electric Co.
Manufacturer of resin	American Cyanamid Co.
Number of resin	Melmac 402
Type of resin	Melamine-Formaldehyde
Composition of resin	1 mol melamine 2 mols formaldehyde 60 per cent solids Solvent: 95 per cent water, 5 per cent butanol Catalyst: none pH: 8.4
Fabric	ECC-11-128
Warp yarn	225-V3: 5 turns per inch (Z) in twisting 4.4 turns per inch (S) in plying
Fill	same as warp
Weave	plain
Thickness	0.007 in.
Weight, raw, of fabric	6.00 oz. per sq. yd.
Thread count of fabric	42 by 32
Tensile strength of warp	250 lb. per in., min.
Tensile strength of fill	200 lb. per in., min.
Number of plies in laminate	62 (estimate)
Lay of laminations	Parallel Laminated
Molding temperature	284 F.
Molding pressure	1250 psi.
Molding time	1 hr.
Temperature when removed from press	room

Test Apparatus; Relative Humidity Measurement:

The apparatus used for the static tension and compression tests and the creep tests at 77 F. has been described previously (2, 3). Also, the construction of the insulated cabinets for creep tests at 39.7 ± 2 F. and 129.6 ± 2 F., the method of controlling humidity in these cabinets by a glycerol-water solution at 50 ± 5 per cent and 50 ± 2 per cent respectively, and the apparatus used in measuring temperature and relative humidity in the cabinets were as described in a previous paper (1) except as discussed below.

It was found that the thin rubber membranes previously used as moisture seals were too permeable. Less permeable seals were constructed of brass washers held in place around the loading rods by spring clips. Grease was used to seal the remaining small annular space.

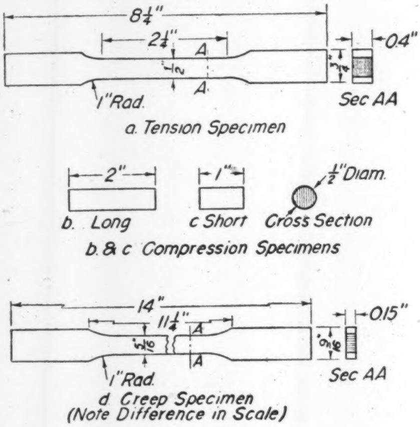

FIG. 1.—Static and Creep Specimens. Section lines indicate plane of laminations.

It was found necessary to draw the air sample at a particular time in the heating or cooling cycle of the cabinets.

Special nomographs were constructed to speed the calculation of relative humidity.

To date, we have not found a satisfactory sensing element for relative humidity which will maintain its calibration during changes from 40 to 77 F. or from 77 to 130 F., and be reasonably free from drift. Methods of measuring the relative humidity which have been tried unsuccessfully since the first report on creep tests with this apparatus included the dew point method, the method of measuring the resistance of a hygroscopic salt and a diffusion method.

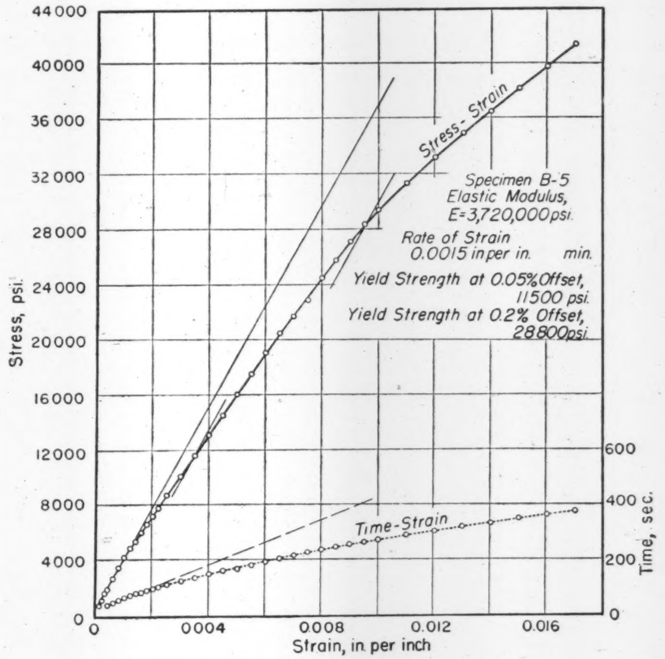

FIG. 2.—Representative Static Tension Test Results for a Melamine-Glass Fabric Laminate.

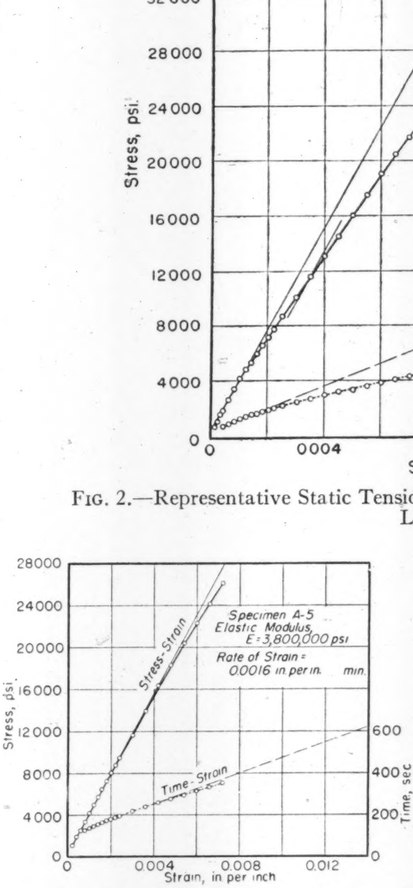

FIG 3.—Representative Static Compression Test Results for a Melamine-Glass Fabric Laminate.

Test Procedure:

The static tests were run at constant rate of head motion and both tension and compression tests were conducted at the same rate of strain (0.0016 in. per in. per min.). The technique used was the same as described previously (**2**, **3**).

The procedure used in conducting the tension creep tests was substantially the same as used in the earlier paper (**1**) except that a longer time elapsed (30 days) from the time the specimens were mounted to the time the loads were applied and a longer time was required (2 days) to complete initiating the series of tests at each temperature.

Results:

Representative stress-strain diagrams are shown in Fig. 2 for static tension tests and in Fig. 3 for static compression tests of typical specimens of the melamine laminate. The ultimate strength, yield strength at 0.05 and 0.2 per cent offset, initial modulus of elasticity and secondary modulus of elasticity in tension and compression are given as averaged values in Table II together with the fatigue strength at 10^7 cycles and average values of specific gravity and Rockwell hardness.

It was observed that the laminate was about 66 per cent stronger in tension than in compression. An abrupt change in slope of the stress-strain curve occurred at about 4000 psi. in tension, but not in compression. The slope of the curve below this point has been called the initial modulus and the slope above the point the secondary modulus.

TABLE II.—A SUMMARY OF THE MECHANICAL PROPERTIES OF THE MELAMINE-GLASS FABRIC LAMINATE.

Specific gravity	1.90
Initial tension modulus of elasticity, psi	3 870 000[d]
Secondary tension modulus of elasticity, psi	2 930 000[d]
Ultimate strength in tension, psi	43 100[d]
Tension yield strength at 0.05 per cent offset, psi	11 300[d]
Tension yield strength at 0.2 per cent offset, psi	27 300[d]
Compression modulus, psi	3 900 000[d]
Ultimate strength in compression, psi	26 100[d]
Compression yield strength at 0.05 per cent offset, psi	[a]
Compression yield strength at 0.2 per cent offset, psi	[b]
Rockwell hardness, perpendicular to laminations	M 119[d]
Fatigue strength at 77 F., 1800 rpm., and 10^7 cycles,[c] psi	14 600

[a] Specimens failed before 0.05 per cent offset was reached.

[b] Specimens failed before 0.20 per cent offset was reached.

[c] Unnotched specimens tested in a rotating-cantilever-beam fatigue machine.

[d] Average of four tests.

The creep and recovery as a function of time are presented in Figs. 4 to 6 for the three test temperatures. The relative humidity and the weight of the control specimen have been included in these figures to aid in interpretation of the data. The change in length of a control specimen which carried no load is also included in these figures.

Only a relatively small amount of creep occurred and it occurred at a decreasing rate approaching zero for this laminate. After the creep data had been extended to about 3500 hr., the loads were removed from the specimens and the recovery of the strain was recorded as shown in Figs. 4 to 6.

There was no significant change in weight of any of the control specimens. However, a very slight downward trend was observed for the 129.6 F. test indicating that the specimen may have been drying out at a very slow rate. It was also noted that the control specimen stretched at 39.7 F. and shrank at 129.6 F., whereas at 77 F. the specimen first stretched and then contracted but did not deviate very far from its initial value throughout the time of testing.

Since the equipment for determining relative humidity was not available to the investigation beyond the time when the recovery tests were initiated at the high and low temperature, no record of relative humidity is available for the last 1000 hr. in Figs. 4 and 6. It is apparent from the decrease in the length of the control specimen at 129.6 F., that the relative humditiy decreased rapidly beyond 3500 hr. Thus the apparent high rate of recovery beyond this point must be corrected for shrinkage in the control specimen. The behavior of the control specimen at 39.7 F. indicates that the relative humidity did not deviate very greatly or that its effect was negligible.

Considering the relative humidity over the first 3200-hr. period, it appears that the control was not as close as desirable. It will be observed that a discontinuity occurs in the data at about 250 hr. for the tests at 39.7 F. (Fig. 4). It was not possible to determine the cause of this disturbance but it was observed that the relative humidity did increase at this time and then slowly returned to a lower mean value. However, the magnitude of the disturbance appeared to be far too great to have been the result of this factor alone. Another possible explanation may have been that the cooling unit ceased to function, permitting the temperature in the test

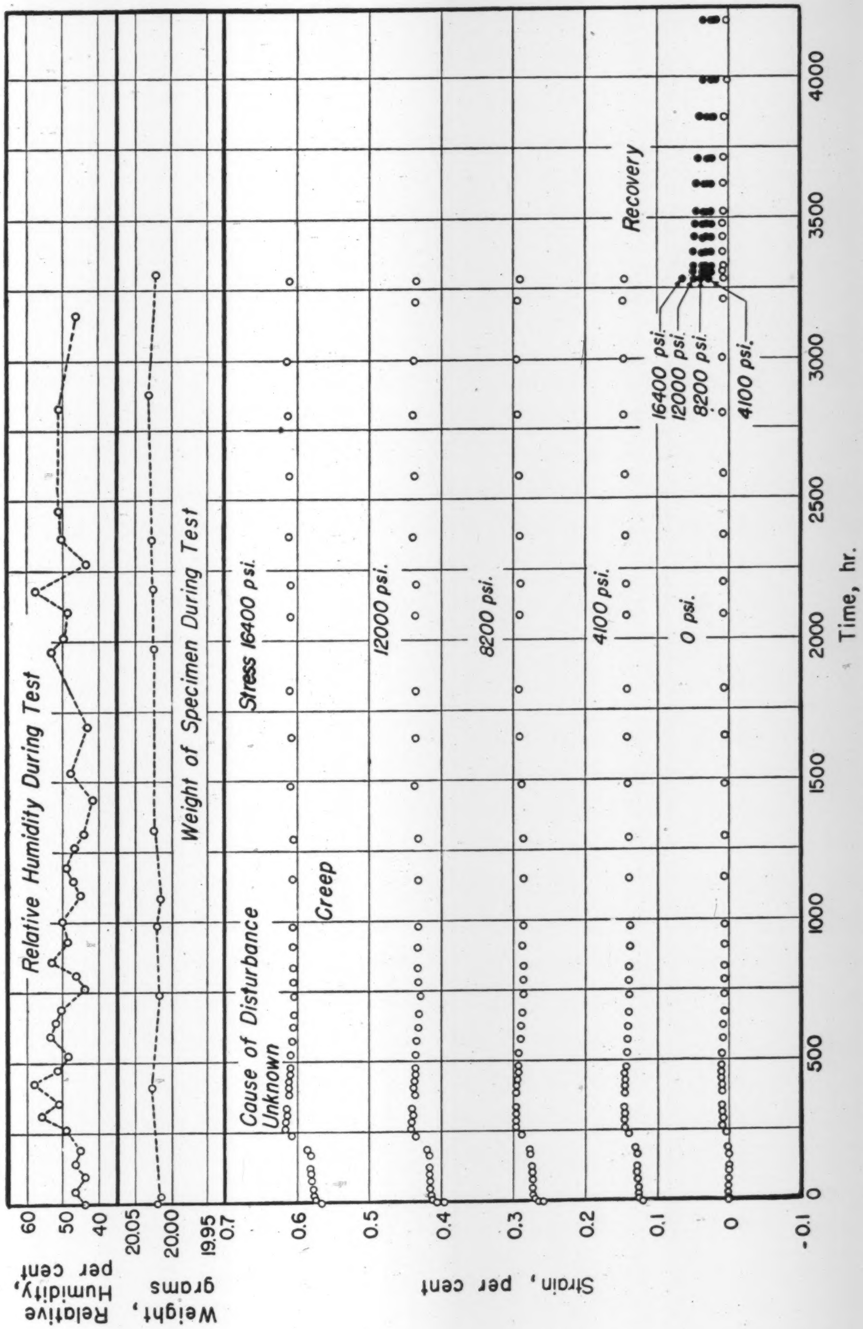

FIG. 4.—Creep and Recovery *versus* Time for Tension Creep Tests at 39.7 F. on Melamine-Glass Fabric Laminates.

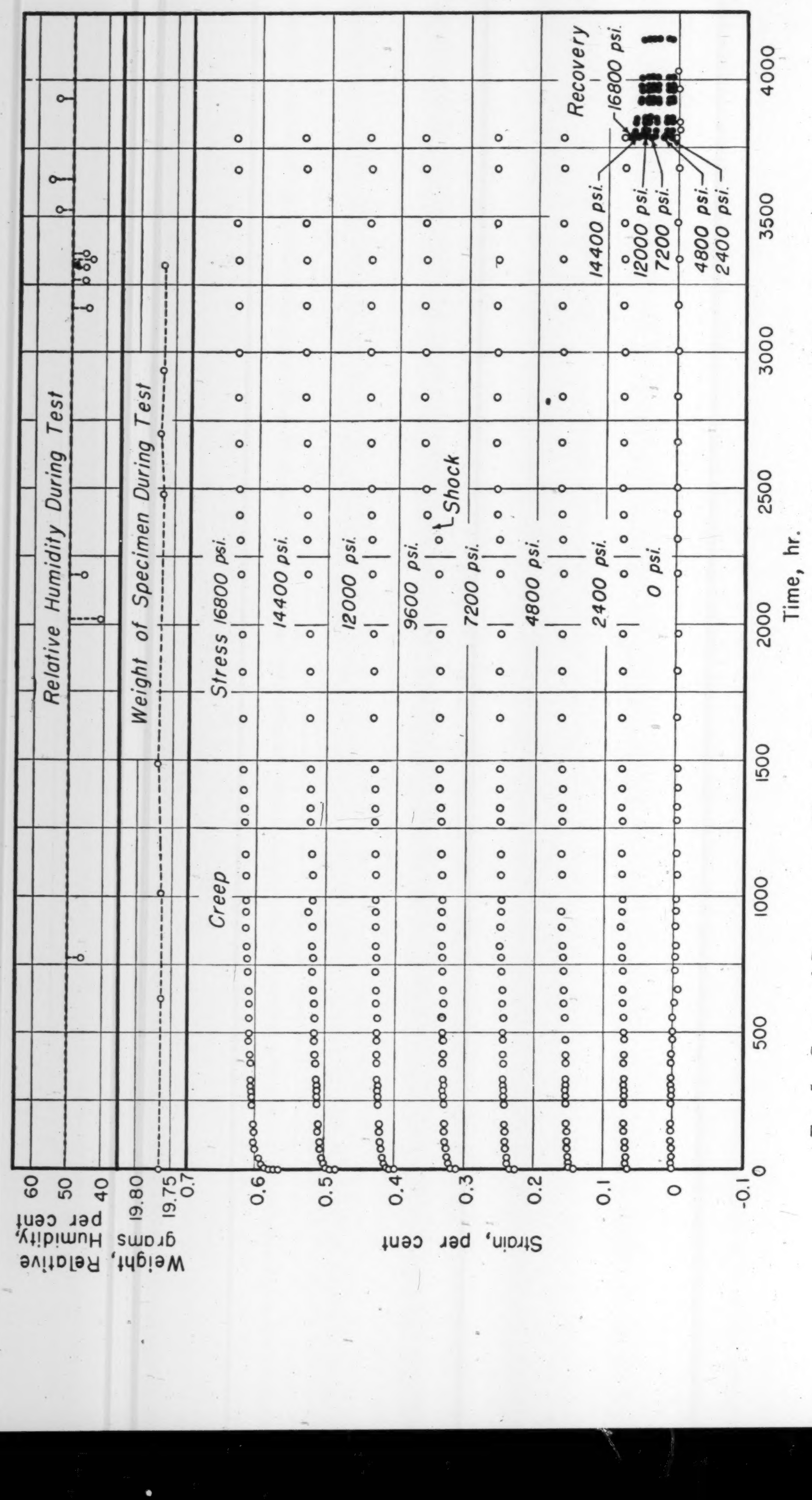

FIG. 5.—Creep and Recovery *versus* Time for Tension Creep Tests at 77 F. on Melamine-Glass Fabric Laminate.

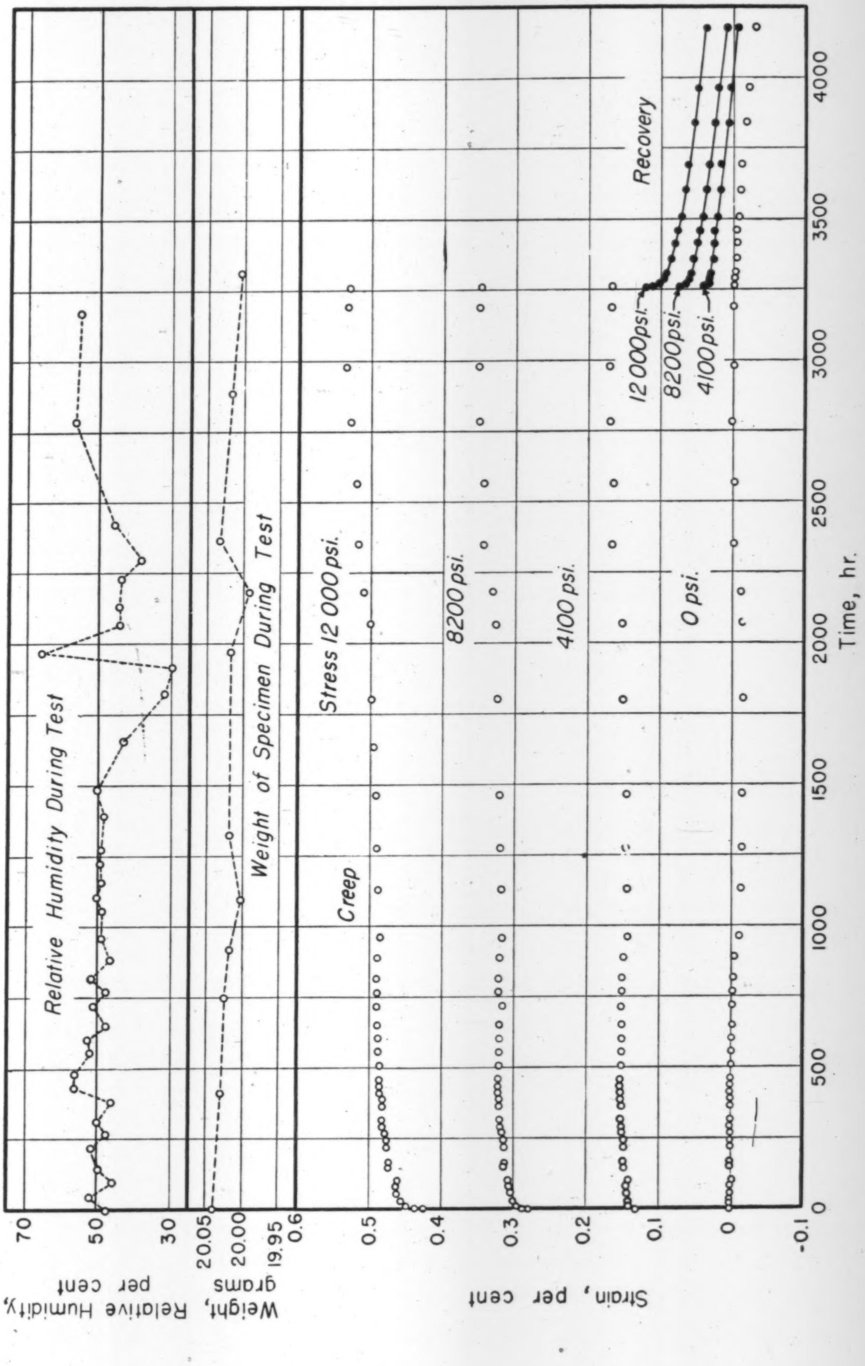

FIG. 6.—Creep and Recovery *versus* Time for Tension Creep Tests at 129.6 F. on Melamine-Glass Fabric Laminates.

chamber to approach room temperature. If this occurred, control was regained automatically before the next check was made since no such trouble was noted.

Since a continuous record of temperature was not available, there was no way to verify this possibility, but an increase in temperature would have resulted in an increase in specimen length which might not have recovered immediately. It was noted also that the data before 250 hr. appeared to line up with the data just beyond 1000 hr.; thus the specimens appear to have recovered from the disturbance in this time interval.

TABLE III.—SUMMARY OF CREEP TEST DATA.

Temperature, deg. Fahr.	Relative Humidity, per cent	Stress, psi.	ϵ_1, Instantaneous Strain at 20 sec., per cent	ϵ_2, Total Strain at 1000 hr., Corrected for Shrinkage, per cent	ϕ_{12}, Increase in Strain (Creepocity) 20 sec. to 1000 hr., per cent	ϵ_0, Coefficient in Eq. (1), Average of Values Computed from ϵ_1 and ϵ_2, per cent	ϵ_3, Total Strain at 3000 hr., Corrected for Shrinkage, per cent	ϵ, Total Strain Computed from Eq. (1) at 3000 hr., per cent	Instantaneous Recovery at 20 sec., per cent	v, Creep Rate at 500 hr., 10^{-7} in. per in. hr.	Creep Rate Computed from Eq. (5) at 500 hr., 10^{-7} in. per in. hr.
39.7	50	4 100	0.1186	0.1310	10.4	0.0618	0.1373	0.1312	0.1194	...	0.184
		8 200	0.2577	0.2787	8.2	0.1323	0.2865	0.2815	0.2522	...	0.395
		12 000	0.3944	0.4249	7.7	0.2035	0.4284	0.4330	0.3858	...	0.608
		16 400	0.5508	0.5983	8.6	0.2853	0.6050	0.6060	0.5453	...	0.851
					8.7 (Avg.)						
77.0	50	2 400	0.0666	0.0805	21.0	0.0363	0.0808	0.0782	0.0649	1.45	0.152
		4 800	0.1449	0.1681	16.0	0.0772	0.1696	0.1667	0.1403	1.95	0.323
		7 200	0.2289	0.2554	11.6	0.1196	0.2639	0.2580	0.2202	1.78	0.500
		9 600	0.3114	0.3402	9.3	0.1611	0.3524	0.3475	0.3013	1.38	0.674
		12 000	0.4004	0.4342	8.5	0.2063	0.4467	0.4450	0.3837	1.62	0.864
		14 400	0.4842	0.5260	8.6	0.2498	0.5403	0.5390	0.4724	2.26	1.035
		16 800	0.5700	0.6212	9.0	0.2918	0.6366	0.6290	0.5532	2.52	1.215
					12.0 (Avg.)						
129.6	50	4 100	0.1285	0.1554	20.9	0.0695	0.1712	0.1568	0.1245	0.79	0.466
		8 200	0.2782	0.3288	18.2	0.1490	0.3528	0.3355	0.2683	1.58	0.997
		12 000	0.4225	0.4985	18.0	0.2260	0.5352	0.5080	0.4080	2.70	1.512
					19.0 (Avg.)						

The "instantaneous" strain (ϵ_1) occurring at the initiation of a creep test was defined as the total strain measured in the specimen at the time at which the load had been fully applied to the specimen for 20 sec. Since a reading was not usually obtained at 20 sec. the 20-sec. "instantaneous" strain was determined by plotting the readings of strain obtained during the first hour of the test as a function of the time in seconds on log-log paper. A straight line was then drawn through the plotted points for each stress, and the strain corresponding to a time of 20 sec. was read from the straight line. This technique was based on the observation that creep data yield nearly linear curves for at least the first few hours when plotted on log-log coordinates (**4**, **5**).

The values for the 20-sec. "instantaneous" strains are presented in Table III. When plotted as functions of stress for the three temperatures, these data form smooth straight curves, similar to the stress-strain curves for the static tension test shown in Fig. 2, and it is possible to obtain a value for the modulus of elasticity for these three temperatures. The moduli of elasticity obtained in this manner from the instantaneous creep *versus* stress relationship were 2,840,000 psi. at 77 F., 2,910,000 psi. at 39.7 F. and 2,740,000 psi. at 129.6 F., and were reasonably close to the secondary tension modulus, 2,930,000 psi. at 77 F.

The total strain (ϵ_2), including the instantaneous strain, was measured for all specimens at 1000 hr., and all strain values were corrected for the change in length of the zero load specimen. The corrected values for the total strain (ϵ_2) appear in Table III. When plotted as functions of stress for the three temperatures they also form smooth almost-straight curves, having a very slight upward curvature, for all three temperatures. The readings for the total strain at 39.7 F. are nearly the same as those at 77 F., probably because of the disturbance in the 39.7 F. creep curves mentioned earlier.

The slope of the creep curves was so small that it was difficult to obtain an accurate determination of the rate of creep. The values measured at 500 hr. are shown in Table III except for tests at 39.7 F. where the disturbance previously noted prevented measurement of the creep rates.

The 20-sec. "instantaneous" recovery of strain on unloading the specimens are listed in Table III. These data indicate that most of the strain (about 97 per cent), which took place instantaneously on loading, was recovered instantaneously on removal of the load after about 3500 hr. under load. The recovery data (not all of which is given on the curves) also show that about 60 per cent of the creep (time-dependent strain) occurring during the 3500 hr. under load was recovered in 2000 hr. following removal of the load. Recovery data available for some of these tests for as long as 7000 hr. show very little additional recovery. This indicates that a small amount of irrecoverable or secondary creep will remain.

Discussion of Results:

The data on total strain yields further evidence in support of the proposition described in reference (2) that the per cent increase in strain from the start of a creep test to a given time is a constant independent of stress. This quantity was called the "creepocity" in the above reference and is given in Table III for the present tests. Except at low stresses where the measurement of creepocity was the least accurate the value was substantially the same for all stresses at a given temperature, but increased with temperature.

As discussed in the reference cited, creepocity, if definitely established for a given class of material, would serve as a convenient measure of the serviceability of the material under conditions of creep. High values of creepocity would denote poor resistance to creep.

Of course the creepocity alone will not define the complete creep behavior of a material even at a given temperature, but it may partially fill the gap existing when a designer has only creep rate as a measure of creep resistance.

For example, if creep behavior of a material can be expressed with reasonable accuracy by an equation of the form

$$\epsilon = \epsilon_0(1 + t^n) \ldots\ldots\ldots\ldots (1)$$

where:

ϵ = strain, total, in. per in.,
ϵ_0 = strain, initial, in. per in.,
t = time, hr., and
n = exponent, dimensionless.

As proposed for some plastics in an earlier paper by the author (5) then the per cent increase in strain, ϕ_{12}, from time t_1 to time t_2 is

$$\phi_{12} = \frac{\epsilon_0(1 + t_2^n) - \epsilon_0(1 + t_1^n)}{\epsilon_0(1 + t_1^n)}$$

$$100 = \frac{t_2^n - t_1^n}{1 + t_1^n}\, 100 \ldots (2)$$

If t_1 is chosen as zero, n may be computed from ϕ by the following:

$$n = \frac{\log_{10} \phi_{12} - 2}{\log_{10} t_2} \ldots\ldots\ldots\ldots (3)$$

or if t_1 is chosen as unity,

$$n = \frac{\log\left(\frac{1}{50}\phi_{12} + 1\right)}{\log t_2} \ldots\ldots\ldots (4)$$

Equation 4 is more useful than Eq. 3 since creep proceeds so rapidly during loading and during the first few minutes that the strain at zero time cannot be determined with sufficient accuracy.

TABLE IV.—CONSTANTS FOR HYPERBOLIC SINE EQUATION.

Temperature, deg. Fahr.	v_0, 10^{-7} in. per in. hr.	σ_0, psi.	ΔH^*, Activation energy, cal. per mole	ΔS^*, Activation Entropy, cal. per mole deg. K	qAl, (Å)^3	n, Time Exponent, dimensionless	E, Modulus of Elasticity from Creep Tests, psi.
39.7	1.27	26 000	4500	−91	43	0.0137	2 910 000
77.0	2.54	35 000	4500	−91	34	0.0186	2 840 000
129.6	5.70	46 000	4500	−91	29	0.0281	2 740 000

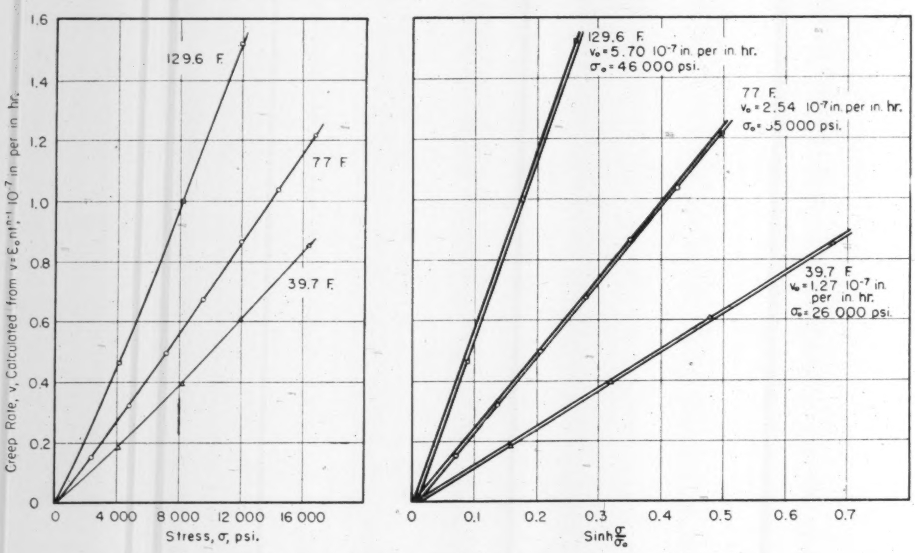

FIG. 7.—The Effect of Stress on the Creep Rate Calculated at 500 hr.

The value of ϵ_0 in Eq. 1 can be determined from tests at each stress by means of measured values of creep rate v at a known time t_2 and the value of n determined as above. The creep rate is given by differentiating Eq. 1 thus:

$$v = \frac{d\epsilon}{dt} = \epsilon_0 n t^{n-1} \quad \text{.........} (5)$$

from which ϵ_0 may be computed.

A second method of determining ϵ_0 is to substitute the value of ϵ at a given time in Eq. 1 and solve for ϵ_0. If the strain ϵ_1 at 1 hr. is used, the relation becomes

$$\epsilon_0 = \frac{\epsilon_1}{2} \quad \text{..............} (6)$$

The procedure of using these relations may be illustrated by the following: From the average creepocity the values of n were computed from Eq. 2 and are given in Table IV. The values of ϵ_0 were then computed by the second method using the value of n and the values of strain at either 20 sec. or 1000 hr. (see Table III). These values of n and ϵ_0 were then substituted in Eq. 1, the strain at 3000 hr. was computed, and the values compared with those measured from the creep tests (see Table III). All but one of the computed values were within 5 per cent of the measured creep.

If, on the other hand, the measured creep rates for tests at 77 F. were used with Eq. 5 to determine ϵ_0, the values of strain at 3000 hr. determined by substitution in Eq. 1 were found to be much too large, due in part to the uncertainty of the measured creep rates and to the fact that Eq. 1 appears not to fit the data exactly.

The values of creep rate at 500 hr. determined by substituting values of n, ϵ_0 (second method), and t in Eq. 5 were much less than those measured from the slope of the creep curves at 500 hr. This discrepancy is due in part to the difficulty of measuring the slope accurately and in part to the fact that Eq. 1 does not fit the data exactly. The computed creep rates are also shown in Table III.

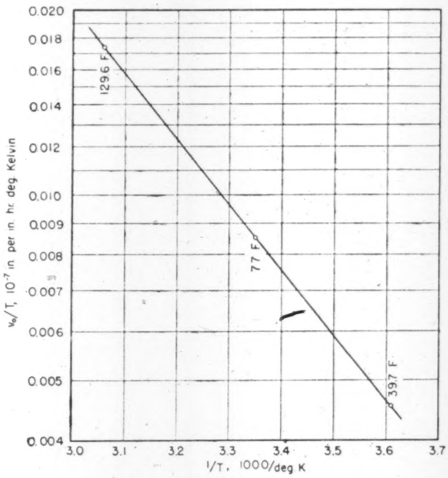

FIG. 8.—Correlation of Cree Rate and Temperature for a Melamine-Glass Fabric Laminate.

It was observed that the computed creep rates were much more consistent in their stress dependence than the measured values, see Fig. 7. Thus it was possible to compare the creep properties of the melamine laminate with the activation energy theory for temperature and stress dependence.

The activation energy theory for creep has been reviewed in previous papers (1, 6) and may be expressed in the following form:

$$v = \frac{4}{3}\frac{\lambda}{L}\frac{k}{h}\, T e^{\frac{\Delta S^*}{R}}\, e^{-\frac{\Delta H^*}{RT}} \sinh \frac{qAl\sigma}{2kT} \quad \ldots (7)$$

where:

v = the creep rate, in. per in. sec.,
T = the absolute temperature, deg. K.,
ΔS^* = the entropy of activation, cal. per mole, deg. K.,
ΔH^* = the energy of activation, cal. per mole,
σ = the tensile stress, dynes per sq. cm.,
k = the Boltzmann constant,
h = the Planck constant,
R = the gas constant
λ, L, A = dimensions of the "unit of flow,"
q = stress concentration factor, and
l = the distance through which the shear stress acts in carrying the unit of flow from the normal to the activated state.

To test the applicability of Eq. 7 with respect to stress dependance the equation may be written as

$$v = v_0 \sinh \frac{\sigma}{\sigma_0} \quad \ldots\ldots\ldots\ldots (8)$$

This relation was found to fit the derived creep rates reasonably well as shown in Fig. 7(*b*). From values of v_0, σ_0 and the absolute temperature T it was possible to calculate the activation energy, ΔH^*, activation entropy, ΔS^*, and product qAl in the same manner described previously (1). See Table IV.

These calculations involved plotting log (v_0/T) *versus* $1/T$ as shown in Fig. 8. At 129.6 F. the creep rate was so nearly a linear function of the stress that v_0 and σ_0 could not be determined precisely as independent quantities, but their ratio

$\frac{v_0}{\sigma_0}$ could be determined. For this reason the value of v_0 was chosen so as to lie on the straight line drawn through the other two points in Fig. 8.

This straight line is in accordance with the requirements of the theoretical equation, Eq. 7. From the slope and intercept of the straight line the values of ΔH^* and ΔS^* were determined and are listed in Table IV together with values of qAl calculated from σ_0 and T by substitution in Eq. 6 of the previous paper (1). It may be mentioned that the activation energy and activation entropy of a composite material such as the present laminate are probably effective values representing the contribution of all components.

An examination of Eqs. 1 and 7 discloses that: the time-sensitivity of creep as expressed by these equations is represented by the exponent n (and hence the creepocity ϕ); the temperature sensitivity of creep is given approximately by the activation energy ΔH^* (when $\frac{qAl\sigma}{kT}$ is small as in the present instance, the approximation is very good); the stress sensitivity of the creep is given by qAl; and the change in degrees of freedom in the structure of the material resulting from creep is given by the activation entropy ΔS^*. That is, a negative value of activation entropy indicates that the creep places certain molecules or fibers in a more strained position than formerly so that these components have fewer degrees of freedom. The net result is a less random distribution of energy in the interatomic or intermolecular bonds.

If the results of creep tests previously reported for canvas laminate (1) are compared with that of the melamine-glass fabric laminate it is found that the time sensitivity was much less for the melamine-glass fabric, as indicated by the creepocity. The average value of the creepocity from 20 sec. to 800 hr. for the canvas laminate (not previously reported) was 47.6, 59.1 and 113.2 per cent at 42.5, 77.0 and 128.4 F. respectively or about 6 times the creepocity from 20 sec. to 1000 hr. for the melamine-glass fabric laminate. Also, the temperature sensitivity as measured by the activation energy was much less for the glass fabric laminate (450 compared to 16,000) the stress sensitivity as measured by qAl was much less (29 to 43 compared to 105 to 622) and the decrease in degrees of freedom resulting from creep as measured by the activation entropy was greater for the glass fabric laminate (-91 compared to -49.6).

It was found that qAl decreased with temperature for the glass fabric laminate as observed also for canvas laminate and one of the 14 metals studies by Kauzmann (7). According to Kauzmann's interpretation this means that for the fiberglas laminate the area in the plane of shear stress of the structural element involved in the flow process decreased with increase in temperature.

A comparison of the present data with that obtained for a glass fabric laminate molded with polyester (**2**, **3**) indicated that the creepocity from 20 sec. to 1000 hr. was 16.4 per cent for the polyester laminate and 12 per cent for the melamine laminate at 77 F. This indicates that the polyester laminate was somewhat more time-sensitive than the melamine laminate.

The time sensitivity of rayon laminate (**1**, **2**) as measured by creepocity was much greater than that of the melamine-glass fabric laminate. The average creepocity from 20 sec. to 800 hr. for the rayon laminate (not previously reported) was 73.5, 82.3, and 88.8 for temperatures of 42.5, 77.0, and 128.4 F. respectively.

It is of interest to note that the creepocity increased with temperature for these three laminates.

Discussion of Creep Testing Technique:

Measurement of strain to 0.0000001 in. per in. is needed for an accurate determination of creep rate at low stresses or moderate temperatures.

In addition to the precise measurement of strain, the problems of providing sufficiently accurate measurement and control of variables such as temperature, moisture content of the specimen, and extraneous vibration, were considered.

Close control of these other variables is required because small changes in any of them result in variations of the strain which may exceed the magnitude of the strain resulting from creep for short intervals of time.

It was not possible for these quantities to be recorded automatically in the tests but had such records been available, it would have aided in the interpretation of the test data. To improve the present testing apparatus, the continuous recording of temperature would offer no serious problem and the relative humidity could be recorded with a sensing element which would either change the resistance of or apply a voltage to an electrical circuit. Here the chief problem that might be encountered, however, would be the drifting of the zero of the sensing element and the auxiliary equipment.

The rapid shrinkage of the glass-fabric laminate at 129.6 F. when the relative humidity was no longer controlled suggests a need for control of relative humidity for tests at still higher temperatures even for the glass-fabric laminate. At such temperatures shrinkage due to loss of moisture content may present a serious problem unless the vapor pressure of the atmosphere surrounding the specimen can be maintained at the same value as the internal vapor pressure of the specimen.

In order to prevent dimensional changes during long-time tests, such as shrinkage of the specimens resulting from changes in moisture content, the following methods might be used in an improved apparatus. The first method involves heating the specimens to the test temperature in a creep testing machine located in a laboratory having a controlled temperature (77 F.) and relative humidity (50 per cent) and maintaining the specimens at this temperature until their lengths stabilize, before the creep tests are started. Then the specimens are tested in creep in the same controlled laboratory. The other method involves adding moisture to the test cabinet or furnace using one of the two methods of detection and correction described below.

The first of these consists of manually or automatically adding moisture to the cabinet in sufficient quantity to prevent the length of a specimen, equipped with an extensometer and carrying no load, from changing. The second method consists of manually or automatically adding moisture to the cabinet in response to an indicating or controlling scale beam mounted within the test chamber in such a way that the beam responds to a change in the weight of a specimen.

The continuous recording of shocks or transient vibrations as well as steady state vibrations of the creep test apparatus could be accomplished by means of suitable vibration measuring and recording instruments capable of detecting low-frequency vibrations.

Summary and Conclusions:

The relation between the total strain at 1000 hr. and the stress appeared to be nearly a linear function for all three temperatures; whereas, for the materials studied in the earlier paper (1), there was a very pronounced curvature.

The creep resistance as indicated by the total strain at 1000 hr. was affected only slightly by temperature.

The per cent increase in strain for a given time interval ("creepocity") during the first 1000 hr. was nearly independent of stress but increased with increasing temperature.

The recovery data indicated that approximately 97 per cent of the 20-sec. instantaneous strain was recovered in the 20-sec. instantaneous recovery following a period of about 3500 hr. under constant load. The amount of irrecoverable strain was small.

The weight of the control specimens at each temperature remained nearly constant as long as the relative humidity was nearly constant.

The length of the zero-stress specimens increased at the low temperature, remained substantially constant at room temperature, and decreased at the high temperature.

The creep was so small that the apparatus was unable to provide data of sufficient accuracy to evaluate the creep rate by measuring the slope of the line. It appeared that both closer control of temperature and humidity and a more sensitive strain measuring instrument than presently available is needed.

Another method of determining the creep rate from constants in an equation for creep $\epsilon = \epsilon_0 (1 + t^n)$ was employed, which gave consistent results.

The activation energy, ΔH^*, activation entropy, ΔS^* product of qAl and time exponent n, were evaluated and their meanings in terms of the properties of the material were discussed.

Acknowledgments:

The authors wish to thank the National Advisory Committee for Aeronautics for financial assistance in connection with the tests at 77 and 130 F. and for permission to publish the data presented. We are also grateful to the Monsanto Chemical Co., Plastics Division, who supplied the laminate for this series of tests.

This project was a part of the work of the Engineering Experiment Station of the University of Illinois, of which W. L. Everitt is Director, in the Department of Theoretical and Applied Mechanics, of which F. B. Seely is Head. The authors are indebted to F. B. Seely for criticism of the paper, and to G. E. Chicoine, H. E. Anderson, J. P. Cedarholm, D. D. Strohbeck, S. C. Hu, and L. H. Shanin for their assistance in collecting and organizing the data presented.

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(7) W. Kauzmann, "Flow of Solid Metals from the Standpoint of the Chemical-Rate Theory," *Transactions*, Am. Inst. of Mining and Metallurgical Engineers, Vol. 143, p. 57 (1941).

DISCUSSION

Mr. V. F. Downing.[1]—Were any of those temperatures, mentioned as high temperatures, anywhere near the cure temperature of the resin itself, or were they all lower? Modified phenol-formaldehyde resins which we have tested at our laboratory seem to exhibit radical changes in physical characteristics at a temperature equivalent to their cure temperature or at temperatures higher than cure temperature.

Mr. W. N. Findley (*author's closure*).—The highest temperature employed in the creep tests reported was 129.6 F., which was considerably lower than the molding temperature of 284 F. However, this project has been reactivated and tests at temperatures up to 400 F. are now in progress on this material. It may be that these tests will show the radical changes Mr. Downing observed.

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[1] Physicist, American Brake Shoe Co., Chemical Lab., Mahwah, N. J.

THE MEASUREMENT OF DYNAMIC MODULUS IN ADHESIVE JOINTS AT ULTRASONIC FREQUENCIES*

By A. G. H. Dietz,[1] P. J. Closmann,[2] G. M. Kavanagh,[2] and J. N. Rossen[2]

Synopsis

A method has been developed to measure, nondestructively, the dynamic modulus of an adhesive in the bond. The dynamic modulus, E, is defined as the stress-to-strain ratio for sinusoidal displacements, and can be expressed as $E = E_1 - iE_2$, where E_1 is the ordinary Young's modulus and E_2 is the viscous modulus representing dynamic losses in the material. Nolle and Westervelt have derived formulas for E_1 and E_2 of a thin adhesive layer joining two cylindrical bars end-to-end. The relations require a knowledge of the resonance properties of the cemented bar and a continuous bar of the same length and material.

The equipment for obtaining the necessary resonance properties, the resonant frequency and half-power bandwidth, consists of three major parts: a driving system to set up longitudinal ultrasonic vibrations in the specimens, a detecting system to indicate the amplitude of vibrations, and a frequency measuring system.

Measurements have been made on stainless steel bar specimens, cemented with a phenol-formaldehyde polyvinyl-butyral adhesive, which have been subjected to various heat treatments. The results of this study indicate that the measured dynamic modulus is useful for selecting from a group of treated specimens those which have deteriorated and have low tensile strength.

The method described is applicable to cemented-bar specimens composed of other types of bars and adhesives, provided that certain conditions pertaining to the derivation of the formulas for the moduli are met. There is a possibility that similar principles can be used to measure the dynamic modulus of reed type specimens composed of sections of lap joint or laminate.

The typical adhesive joint is a thin and narrowly restricted region binding together two relatively massive bodies. While the joint is performing its function, it is almost impossible to observe; once torn apart to be observed it is beyond further use. This natural inaccessibility has impeded inquiry into the essential characteristics of such joints and has made their inspection prior to actual use very difficult. The powerful experimental method of examination prior and subsequent to a desired treatment has been inapplicable to studies in this field; and the best that has been possible has been the statistical comparison of fragmentary experiments on separate sets of specimens, a comparison which is hindered by the difficulties in securing reproducible specimens. Improvement in this situation requires the invention of nondestructive test methods.

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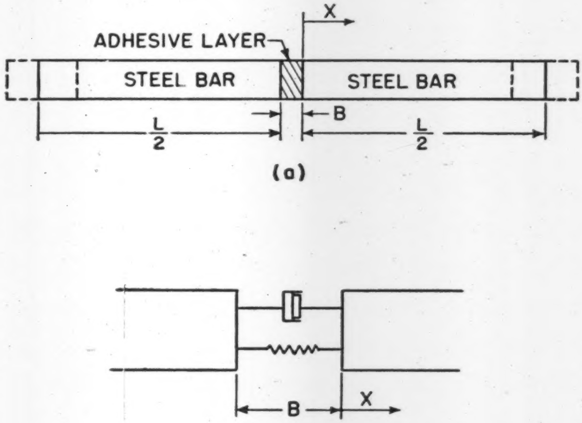
* Presented at the Fifty-third Annual Meeting of the Society, June 26–30, 1950.

[1] Professor of Structural Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2] Staff, Division of Industrial Cooperation, Massachusetts Institute of Technology, Cambridge, Mass.

An attempt to develop a nondestructive test for adhesives must center at first on the possibilities of examining a joint, or of measuring some of its properties without destroying the joint. The experimental method presented here, based on theoretical considerations by Nolle and Westervelt,[3] has led to successful nondestructive measurements of the dynamic modulus of the adhesive layer. The possibility of using these measurements for a nondestructive quality test is explored by means of experiments on heat-induced deterioration.



(b) RETARDED SPRING MODEL OF ADHESIVE LAYER

FIG. 1.—Cemented-Bar Specimen.

DYNAMIC MODULUS AND CALCULATION FROM RESONANCE MEASUREMENTS

Resonance measurements made on cemented-bar specimens lead to the determination of a complex modulus, composed of an elastic and a viscous part, which is considered to be the stress-to-strain ratio for sinusoidal variations. This concept may be visualized by considering the simplified representations of the longitudinally vibrating specimens employed as shown in Fig. 1.

Figure 1(*a*) is a sketch of a specimen with the thickness of the joint greatly exaggerated; the dotted lines indicate the vibrations of the ends of the rod. Figure 1(*b*) depicts the adhesive layer as a simple retarded elasticity model with parallel spring and dashpot. The stress resulting from an extension of the adhesive layer by a distance x has elastic and viscous components:

$$\frac{F}{A}\text{ elastic} = \frac{-kx}{b} \quad\text{and}\quad \frac{F}{A}\text{ viscous} = \frac{-\gamma}{b}\frac{dx}{dt}.$$

Here F is force, A is cross-sectional area, k is the spring constant, γ is the intrinsic viscosity coefficient, and b is the thickness of the cement layer. The total stress is then:

$$\frac{F}{A} = \frac{-kx}{b}\frac{-\gamma}{b}\frac{dx}{dt}.$$

When the bar is vibrating longitudinally at angular frequency ω, the extension at time t may be taken to be:

$$x = Re\{Be^{-i\omega t}\}.$$

Here B is the maximum amplitude of vibration. *Re* indicates that only the real part of the complex expression within the brackets is considered.

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[3] A. W. Nolle and P. J. Westervelt, "A Resonant-Bar Method for Determining the Elastic Properties of Thin Lamina," *Journal of Applied Physics*, Vol. 21, No. 4, April, 1950.

Then:

$$\frac{dx}{dt} = Re\{-i\omega B e^{-i\omega t}\}$$

and

$$\frac{F}{A} = Re\left\{\frac{-B}{b}[k - i\gamma\omega]\, e^{-i\omega t}\right\}.$$

The consideration of the dynamic modulus arises by taking $k = E_1$, the elastic modulus, and $\gamma\omega = E_2$, the viscous modulus. We then have:

$$\frac{F}{A} = Re\left\{\frac{-B}{b}[E_1 - iE_2]\, e^{-i\omega t}\right\}.$$

Since the strain is $Re, \left\{\frac{Be^{-i\omega t}}{b}\right\}$ the quantity $(E_1 - iE_2)$ may be considered formally to be a "complex modulus," the magnitude of whose vector is $\sqrt{E_1^2 + E_2^2}$. For the simple physical picture considered here, E_2 is a function of frequency, and in general both parts of the dynamic modulus would be found to vary with frequency. The variation of E_1 with frequency is a result of the oversimplified mathematical treatment and not of a lack of fundamental significance in the measurements.

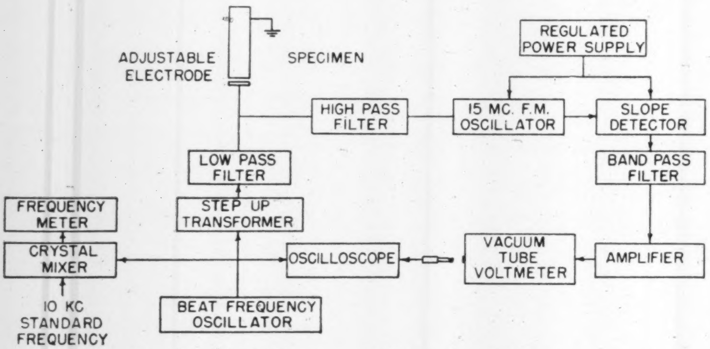
ADJUSTABLE ELECTRODE
SPECIMEN
REGULATED POWER SUPPLY
HIGH PASS FILTER
15 MC. F.M. OSCILLATOR
SLOPE DETECTOR
LOW PASS FILTER
BAND PASS FILTER
FREQUENCY METER
STEP UP TRANSFORMER
CRYSTAL MIXER
OSCILLOSCOPE
VACUUM TUBE VOLTMETER
AMPLIFIER
10 KC STANDARD FREQUENCY
BEAT FREQUENCY OSCILLATOR

FIG. 2.—Block Diagram of Acoustical Apparatus.

The measurements to be considered in this work concern the amplitude-frequency relation of the complete specimen in forced longitudinal vibration at ultrasonic frequencies. In particular, measurements are made of the frequencies at which the driven specimen vibrates with half the energy that it has at resonance; these are the half-power frequencies ν_{low} and ν_{high} of the system. The arithmetic average of these two frequencies is the resonant frequency, ν_1, and their difference is the half-power bandwidth, $\Delta\nu_1$.

For the cemented-bar system described here Nolle and Westervelt[3] have derived the following formulas for E_1 and E_2:

$$E_1 = 4\frac{\rho l}{g_e} b\, \nu_0^2 \frac{\nu_1}{\nu_0 - \nu_1}$$

$$E_2 = \frac{E_1}{2}\frac{\Delta\nu_1 - \Delta\nu_0}{\nu_0 - \nu_1}$$

Here ρ is the density of the bar material, g_c is a consistency factor, and $\frac{l}{2}$ is the length of each rod comprising the cemented specimen. ν and $\Delta\nu$ are the resonant frequency and bandwidth of the resonance peak, respectively. The subscript 1 refers to the resonance properties of the cemented bar, and the subscript 0 refers to the resonance properties of a continuous bar of length l and of the same material.

In the derivation of these formulas it

was assumed that $b \ll l$, the cross-sectional area of the adhesive layer is equal to that of the bar, the ratio $\frac{\nu_1}{\Delta\nu_1}$ is large compared with unity, and that $\frac{\nu_0 - \nu_1}{\nu_0} \ll 1$. If $\frac{\nu_0 - \nu_1}{\nu_0}$ is less than 0.3 the expression for E_1 is accurate to within, 10 per cent. These assumptions have been fulfilled for the measurements to be described here.

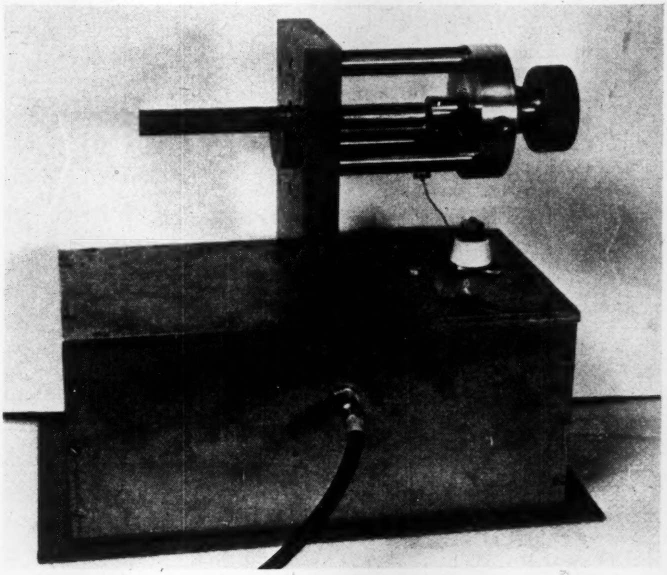

FIG. 3.—Specimen in Mount.

EXPERIMENTAL

The complex modulus of the thin adhesive layer is to be found by comparing the resonance properties of the specimen with similar properties for a continuous bar. In order to measure the resonance properties, the specimen is set into longitudinal vibration by means of an electrostatic driving force. The variation of capacitance between the end face of the bar and a stationary electrode gives a measure of the amplitude of vibration. As the frequency of vibration is varied, the amplitude is measured relative to its value at resonance to provide the desired information on resonance properties.

The apparatus is similar to that of Bordoni[4] and, functionally, consists of three parts: a driving system to set up ultrasonic vibrations in the specimens, a detecting system to indicate the amplitude of the resulting vibrations, and a frequency measuring system. A block and line diagram of this apparatus is shown in Fig. 2.

The driving system consists of a beat frequency oscillator having a frequency range of 20 to 20,000 cycles per sec., a ten-to-one step-up transformer, and a low pass filter. The purpose of the transformer is to step up the output voltage of the driving oscillator to a level where it can cause the rod to vibrate. The low-pass filter, with a cut-off frequency of approximately 50 kc., is so arranged as to prevent the output of a 15-megacycle oscillator in the detecting system from interacting with the driving system. An electrostatic drive force appears across an air gap between the end face of the specimen and an adjustable electrode on the specimen holder (shown in Fig. 3). The specimen rests on three contact points straddling the adhesive joint, which also serve to keep the specimen at ground potential. For each half-cycle of the driving signal, an attractive force results, so that the bar vibrates at twice the frequency of the driving oscillator. No provision is made in the present apparatus for temperature control, since slight variations in room temperature do not affect the results appreciably.

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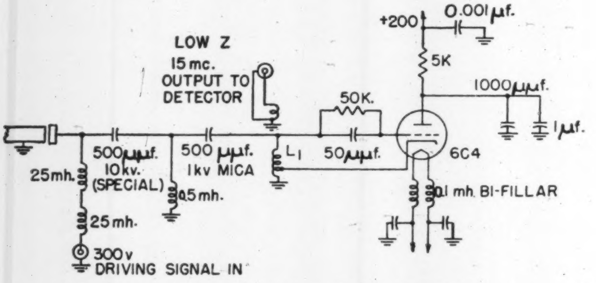
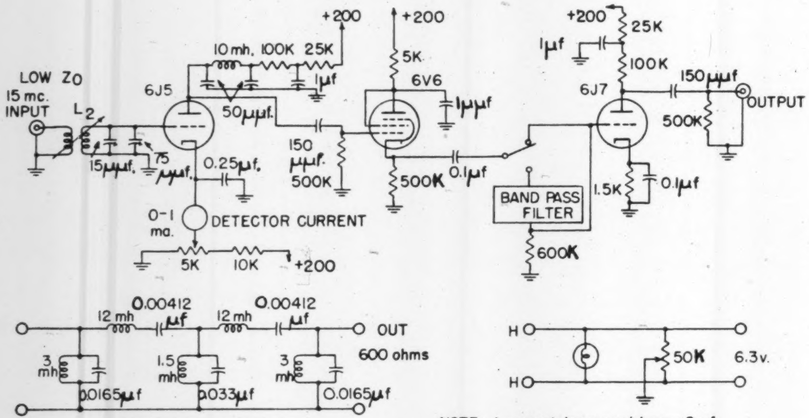
[4] P.G. Bordoni," MetodoElectroacustica per Ricerche Sperimentali Sulla Elasticita," *Nuova Cimento*, Vol. 4, No. 3-4 (1947).


+200
0.001μf.
5K
1000μμf.
1μf.
LOW Z
15 mc.
OUTPUT TO
DETECTOR
50K.
500μμf.
10kv.
(SPECIAL)
500 μμf.
1kv MICA
L1
50μμf.
6C4
25mh.
.5mh.
.1 mh. BI-FILLAR
25mh.
300v
DRIVING SIGNAL IN

NOTE: L1 must have a minimum Q of the order of 100.
Special capacitor (500μμf., 10kv.) must be chosen so that the capacitance remains constant with applied driving voltage.

FIG. 4.—15-Megacycle Frequency Modulated Oscillator.


LOW Zo
15 mc.
INPUT
L2
15μμf.
75 μμf.
6J5
10 mh. 100K 25K
+200
1μf
50μμf.
0.25μf.
150 μμf.
500K
0-1 ma.
DETECTOR CURRENT
5K 10K +200
+200
5K
6V6
1μμf
500K
0.1μf
+200
25K
1μf
100K
6J7
150μμf
OUTPUT
500K
1.5K
0.1μf
BAND PASS FILTER
600K
12 mh
0.00412 μf
12 mh
0.00412 μf
OUT
600 ohms
3 mh
.0165μf
1.5 mh
.033μf
3 mh
0.0165μf
BAND PASS FILTER
16-32 kc.
H
H
50K
6.3v.

NOTE: L2 must have a minimum Q of the order of 200.

FIG. 5.—Detector Assembly.

The capacitance between the end of the rod and the face of the electrode is the tuning capacitance in a 15-megacycle oscillator. As the specimen vibrates longitudinally, the distance between the specimen and the electrode—and hence the capacitance—varies, modulating the frequency of the oscillator. This variation is of a small order of magnitude so that the change in capacitance is linear with the change in distance. The high pass filter in the detecting system, with a cut-off frequency of the order of one megacycle, prevents the driving system output from interacting with the 15-megacycle oscillator. The output of the 15-megacycle oscillator is coupled to a

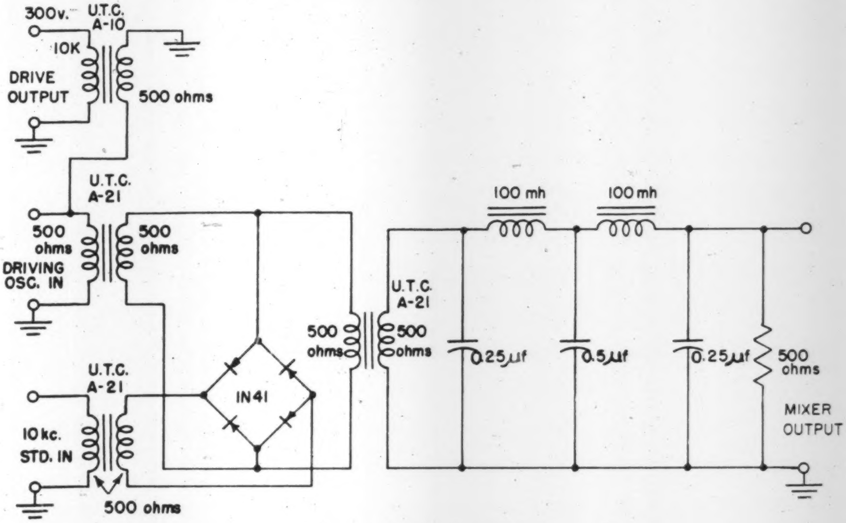

300v.
U.T.C.
A-10
10K
DRIVE
OUTPUT
500 ohms
U.T.C.
A-21
500
ohms
DRIVING
OSC. IN
500
ohms
U.T.C.
A-21
10 kc.
STD. IN
500 ohms
IN41
U.T.C.
A-21
500
ohms
500
ohms
100 mh
100 mh
0.25 µf
0.5 µf
0.25 µf
500
ohms
MIXER
OUTPUT

FIG. 6.—Mixer Assembly.

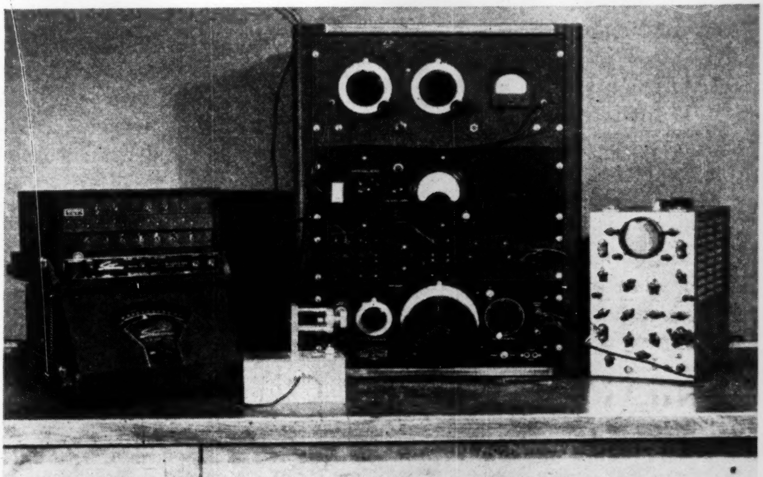


FIG. 7.—Acoustical Apparatus.

slope detector which has a high-Q tuned circuit in order to give the desired sensitivity. This detector converts the frequency modulation of the oscillator output into a detected amplitude variation. The detector output is then fed into a band pass filter (16 to 32 kc.) to improve the signal-to-noise ratio, and the signal is amplified in a resistance coupled amplifier having a gain of approximately 22 decibels. The amplifier output can be indicated on either the oscilloscope or the vacuum tube voltmeter.

The frequency measuring system consists of a crystal mixer to combine the output of the beat frequency oscillator with a 10-kc. standard frequency and a stroboscopic frequency meter to measure the resulting sum or difference frequency.

Circuit diagrams for the 15-megacycle oscillator, the detector, and the crystal mixer are shown in Figs. 4, 5, and 6. Figure 7 is a photograph of the entire apparatus.

When measurements are made on a specimen, it is mounted in the holder shown in Fig. 3; and the electrode adjustment and detector tuning are set for linear operation. The driving oscillator output is increased and the frequency varied to obtain a maximum signal on the oscilloscope. The oscilloscope is then disconnected; and the detector output is indicated on the vacuum tube voltmeter, allowing more accurate adjustment of the driving frequency to the maximum response. The oscillator frequency is finally varied to give an output drop of three decibels on either side of resonance, and these half-power frequencies are measured on the stroboscopic frequency meter. The average of these frequencies is the resonant frequency, and their difference is the half-power bandwidth. From these measurements and other physical properties of the specimen, the moduli can be calculated by use of the applicable formulas.

APPLICATION OF ACOUSTIC METHOD TO STUDY OF HEAT-INDUCED DETERIORATION

The acoustic technique described here was developed to facilitate research in a program to study deterioration in adhesives. It has been useful in measuring the dynamic modulus in specimens prepared from stainless steel rods cemented with a phenol-formaldehyde polyvinalbutyral adhesive that have been subjected to various heat treatments.

In this investigation, sets of twelve specimens are prepared, and acoustical measurements are made on the whole set at room temperature. Six specimens are retained as control specimens for tensile strength measurement, while the remaining six are subjected to various deterioration treatments. Acoustical measurements are made at room temperature at the end of the treatment and, in many cases, during intermediate stages of the tests. Finally the treated specimens are subjected to a tension test.

In order that the measured changes in acoustical and tensile measurements be of significance, it is necessary to have specimens which can be produced easily in the laboratory and which are fairly constant in their initial properties. As a result of an intensive investigation, the following procedure has been adopted for producing specimens:

> The metal rods to be joined are cleaned by removing the old adhesive layers by soaking in acetone and peeling with a flat blade. Adhesive solution is then applied to the ends of the rods with a dropper, and the rods are placed in a closed container in which the air is solvent saturated and are allowed to stand for 24 hr. Another 24 hr. drying is allowed with the container cover opened slightly to allow circulation of air. The rods are finally dried for 2 hr. in an oven heated to 80 C. After drying, the rods are placed in a jig designed to produce 12 specimens with a joint thickness of approx-

imately 0.004 in. The entire assembly is then cured for 45 min. in an oven at 200 C.

This procedure has given satisfactory results with the phenol-formaldehyde polyvinal-butyral resin used to this date. For a different adhesive, modifications may be necessary.

A series of tests was first made to determine the effect of prolonged heating at various temperatures on the adhesive joints. Three sets were treated by being supported in an oven heated to 200 C., 220 C., and 240 C. for 2-hr. periods for a total of 10 hr. After each 2-hr. period the specimens were removed, cooled to room temperature, and acoustically measured.

The effects of heating on the elastic and viscous parts of the dynamic modulus of the joints are shown graphically in Figs. 8 and 9, respectively. Each point is the average modulus for the six treated specimens of each set as measured after each 2-hr. heating period. In Table I the accompanying strength changes are tabulated.

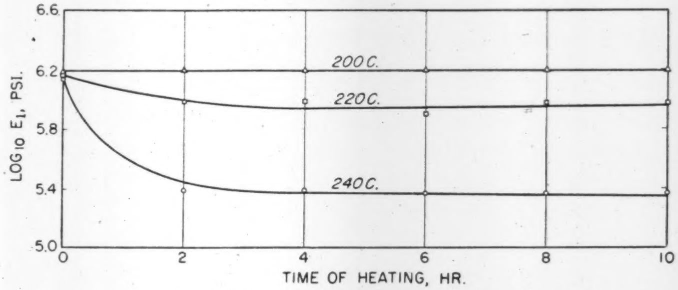


Fig. 8.—Effect of Heating on Elastic Modulus.

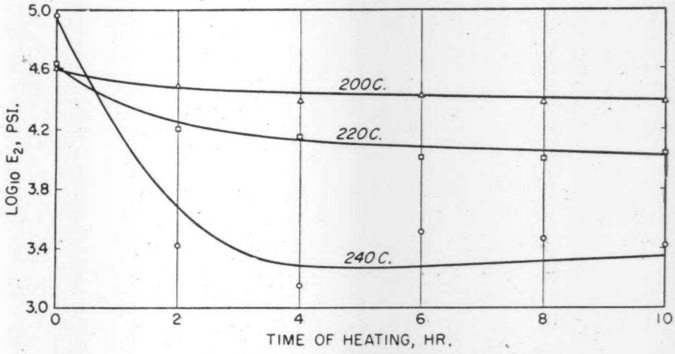


Fig. 9.—Effect of Heating on Viscous Modulus.

TABLE I.—STRENGTH CHANGES AFTER HEATING.

Temperature of Heat Treatment, deg. Cent.	Average Tensile Strength Untreated Specimens, psi.	Average Tensile Strength After 10 hr. Heating, psi.
200	4830	8420
220	6580	5240
240	4710	1430

A comparison of the tensile strengths of the blanks and the specimens treated for 10 hr. indicates that heating at 200 C. causes a strength increase, and heating at 220 C. causes a slight decrease of tensile strength, while heating at 240 C.

causes a serious weakening. From Figs. 8 and 9 it appears that the effects of heating, at least upon the dynamic moduli, occur in the early part of the heating cycle and then level off. This indication from the acoustic method made it seem desirable to examine the early part of the heating cycle. It is to be noted that the results of the tension tests alone could give no indication of the point in the treatment at which deterioration began. With the use of tension tests alone, rather than the nondestructive acoustical method, a great many more tests would have been necessary to obtain the same final indication.

TABLE II.—EFFECT OF HEATING ON DYNAMIC MODULUS AND TENSILE STRENGTH.

	E_1, psi.	σ[a]	E_2, psi.	σ[a]	Tensile Strength, psi.	σ[a]
TREATMENT: HEATED FOR 2 HR. IN 200 C. OVEN						
Blanks (avg. of 5)	797 000	43 000	53 000	17 700	4900	780
Original (avg. of 5)	969 000	310 000	80 800	55 000		
Treated (avg. of 5)	1 035 000	448 000	23 450	21 700	6150	1970
TREATMENT: HEATED FOR 2 HR. IN 240 C. OVEN.						
Blanks (avg. of 6)	1 185 000	262 000	71 000	29 500	4550	590
Original (avg. of 6)	1 004 000	142 000	56 300	23 600		
Treated (individual specimens)	162 700		1 540		1580	
	198 100		1 860		1630	
	288 200		2 850		2400	
	294 800		3 700		1820	
	[b]		[b]		6050	

[a] Represents standard deviation uncorrected for sample size.
[b] Calculation could not be made, $\nu_1 > \nu_0$.

TABLE III.—EFFECT OF HEATING ON DYNAMIC MODULUS AND TENSILE STRENGTH.
Treatment: heated to 230 C. in 240 C. oven (63 min.).

	E_1, psi.	σ	E_2, psi.	σ	Tensile Strength, psi.	σ
Blanks (avg. of 6)	790 000	69 000	50 840	5 300	5250	1280
Original (avg. of 6)	830 000	248 000	33 150	12 000		
Treated (individual specimens)	124 000		1 370		1090	
	119 000		1 620		[a]	
	153 000		1 570		410	
	279 000		3 430		1090	
	975 000		26 010		5720	
	1 005 000		23 530		5900	

[a] Broken in handling.

The next series of tests was made to determine whether the predictions as to the occurrence of changes during the first heating period were correct. Sets were heated for 2 hr. in an oven at 200 C. and at 240 C. These results are given in Table II.

These results confirmed the predictions made by use of the acoustic measurements in the previous series of tests. The changes taking place during prolonged heat treatment occurred early in the heating period. The set heated in the 200 C. oven increased in strength, while the average strength of the set heated in the 240 C. oven decreased.

Considering the results of this series, in particular the definite deterioration in the 240 C. heating cycle, a further investigation of the initial portion of this cycle seemed to be desirable. In this series of tests, specimens were placed in a 240 C. oven with thermocouples attached and the heating curve for this treatment was obtained. Sets were then heated to various points along this curve and measurements made to determine at what point within this short period changes occurred which caused deterioration. Sets heated to 200 C. and 220 C. in the 240 C. oven showed no change in the first case, and slight strength increase in the second. When heated to 230 C. in the 240 C. oven, however, a change was noted, as is shown in Table III.

TABLE IV.—EFFECT OF HEATING ON DYNAMIC MODULUS AND TENSILE STRENGTH.
Treatment: heated to 240 C. in 240 C. oven (110 min.).

	E_1, psi.	σ	E_2, psi.	σ	Tensile Strength, psi.	σ
SET NO. 1						
Blanks (avg. of 6)	816 000	135 000	49 900	10 000	4 380	580
Original (avg. of 6)	932 000	72 000	59 000	12 700		
Treated (individual specimens)	94 000		620		700	
	111 000		40		710	
	99 000		1 040		725	
	744 000		12 800		7 060	
	869 000		9 240		9 150	
	886 000		16 900		10 500	
SET NO. 2						
Blanks (avg. of 6)	886 000	81 000	24 100	3 900	6 310	400
Original (avg. of 6)	970 000	81 000	28 000	2 700		
Treated (individual specimens)	211 000		1 120		905	
	121 000		780		45	
	649 000		8 220		6 420	
	852 000		11 510		9 500	
	946 000		12 650		9 590	
	877 000		12 730		9 050	

Of the six treated specimens, four were deteriorated, while two were slightly stronger than the blanks. It seemed evident that deterioration began in the range from 220 C. to 230 C. The results obtained on two more sets of specimens which were heated for 110 min. in a 240 C. oven are summarized in Table IV. In this period the specimens attained a temperature of 240 C.

Table IV indicates that, in spite of receiving identical treatment, deterioration occurred in some specimens while a marked increase in tensile strength occurred in others. It can be noted, in these cases, that from the moduli of the treated specimens, one can predict whether any individual specimen will be satisfactory or poor in tensile strength.

DISCUSSION OF RESULTS ON HEAT-TREATED SPECIMENS

In discussing the results of the experiments it will be convenient to consider the observations on the change of strength with heating and then to relate the change in acoustic properties to these strength variations.

The results on the specimens that were heated at 200 C. for periods as long as 10 hr. show that there has been a consistent increase in strength. Specimens heated to 220 C. for 2 hr. have also increased in strength. Longer heating at 220 C. or heating at higher temperatures has caused deterioration in some cases, with the likelihood of deterioration increasing with the severity of the treatment. In instances where some of the specimens in a set deteriorate there is remarkable difference in the properties of various duplicate specimens so that they are clearly separated into two groups on the basis of strength. Some of them are found to have increased notably in tensile strength above the average value of the blanks from the same set, while others are found to have deteriorated markedly. This effect is clearly demonstrated in Fig. 10 where a distribution of number of specimens is plotted against strength for the sets in which some specimens deteriorated. The upper plot shows the separation observed in the case of treated specimens. It will be noted that there are no treated specimens left in the region of maximum probability for the blank specimens; that there is a distribution of undeteriorated specimens over a rather broad strength range; and that there is a concentration of specimens with very poor strength.

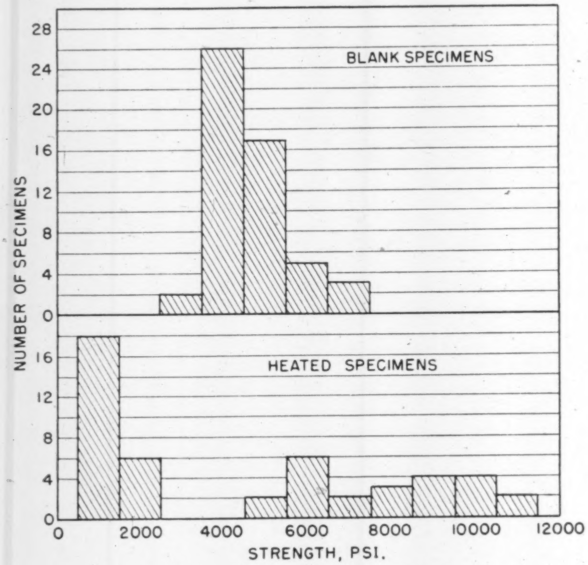


FIG. 10.—Strength Distribution of Specimens.

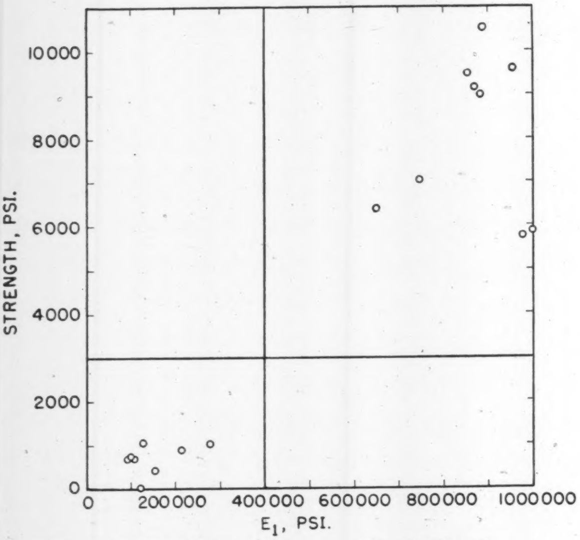


FIG. 11.—Tensile Strength *versus* Elastic Modulus.

The acoustic measurements show that

there is a relation between the strength changes and changes in the dynamic modulus. This can be seen in Figs. 11 and 12, where tensile strength is plotted *versus* the elastic and viscous moduli, respectively. Since E_2 is small compared to E_1, the dynamic modulus is essentially equal to E_1; and hence Fig. 11 may be taken to be a plot of tensile strength *versus* dynamic modulus. For the specimens that have not deteriorated the elastic moduli have not changed much from the values before treatment, while the viscous moduli have decreased somewhat from their original values. The specimens that deteriorate show a marked drop in both moduli; E_1 may decrease to as little as one-tenth of the original value; E_2 drops below 8000 psi. These changes are so characteristic that the specimens which are weak in strength may be selected reliably from the group on the basis of the acoustic test and before the destructive strength tests are made. Figures 11 and 12 illustrate this relation, indicating particularly the separation into two distinct groups.

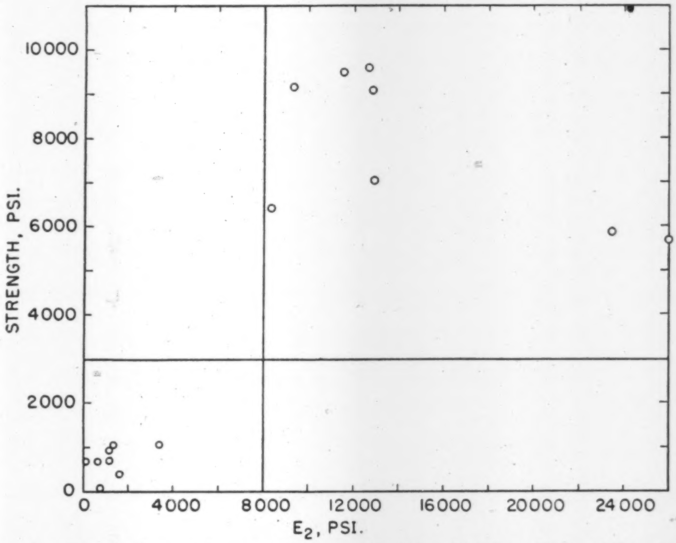


FIG. 12.—Tensile Strength *versus* Viscous Modulus for Heat-Treated Specimens.

In considering the cause of the peculiar changes noted in these joints, it seems likely that they arise from the superimposed or successive action of two separate influences. It is not too hard to rationalize the increase in strength observed in the specimens that did not deteriorate; the increase at 200 C. with extended treatment, and the more rapid increase at the higher temperatures, fits in with the expected nature of a chemical change such as continued cure. This is borne out by a high degree of stiffness (indicated by a high elastic modulus) and lower internal damping (indicated by a low viscous modulus). The sudden decrease in strength and modulus on more drastic heating is considerably harder to explain. It may be due to thermal stresses set up by the unequal expansion of metal and adhesive which result in sudden internal breaking on a small but not molecular scale, or it may be due to a molecular decomposition accompanying a large degree of overcure. An approach to the differentiation of such influence is being made by studies on solid specimens of adhesive material; if these do not show the effect, the influence of restraint by the adherend is indicated; if they do, the answer is almost certainly in the material itself.

APPLICABILITY OF THE ACOUSTIC METHOD

The results of the work described herein indicate that a method has been developed for measuring, in the bond, the dynamic modulus of an adhesive. Depending upon the constraining effect of the adherend, the measured modulus may have a value between a true elastic modulus and a bulk modulus. The constraining effect is dependent upon the ratio of moduli of the adhesive and adherend and the bond thickness-to-diameter ratio. For very small values of thickness-to-diameter ratio, the modulus will be close to a bulk modulus. With higher values the lateral constraint will decrease, and the measured modulus will decrease towards the elastic modulus. In this investigation the adherend was very rigid compared to the adhesive, and, with small thickness-to-diameter values, ranging from 0.006 to 0.03, it is believed that the moduli measured are bulk moduli.

For the adhesive-adherend system employed in this work, the measured dynamic modulus can be used to detect deteriorated specimens from a group of specimens which have been subjected to heat treatment. This is significant, because the method offers possibilities for use as a nondestructive quality test, with applications to other adhesive-adherend systems as well. The specimens employed must be of the cemented-bar type and should be designed so that the conditions mentioned in connection with the formulas for E_1 and E_2 are satisfied. It may be possible to use glass and certain types of plastics with low internal damping as bar material. The restrictions on configuration make the method at present better suited to an experimental technique for measuring adhesive properties, rather than as a production control test where items to be tested would probably not conform to these restrictions. There is some possibility of applying similar principles to measure the dynamic properties of reed type specimens made from a section of lap joint or laminate.

Acknowledgments:

The research with which this report is concerned is being carried out at the Massachusetts Institute of Technology under the sponsorship of the Army Ordnance Department. The authors gratefully acknowledge the assistance given by P. G. Bordoni upon whose apparatus the present one is modeled, and by A. W. Nolle, P. J. Westervelt, and other members of the M. I. T. Acoustics Laboratory who worked with the theoretical basis for the measurements. H. P. Meissner has maintained a continued interest and has offered valuable suggestions throughout the course of the work. The authors also wish to acknowledge the assistance of the A.S.T.M. Advisory Committee for Ordnance Project TB4-601 which committee made a number of helpful suggestions. Credit belongs to Mr. Milton S. Cohen for the modification and construction of the electric apparatus, and to Mr. Arthur A. Schlicting for performing much of the laboratory work.

DISCUSSION

MR. FRANK W. REINHART.[1]—I am familiar with this work and I feel that it is the only real approach at the present time to a fundamental measure of adhesion. In all the other mechanical methods we have used, the method of test introduces certain factors which detract from the basic adhesion value. My concept of adhesion is that there is a basic adhesion value in any particular system, but that there are a lot of factors inherent in the method of test responsible for the value obtained, particularly in mechanical tests. Also, the design of the test apparatus affects the value. The result is that when you make a measurement you are not measuring adhesion, but you are measuring the difference between these characteristics and the basic adhesion.

I think the measuring of the dynamic modulus is the only way we have to determine basic adhesion values at the present time.

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[1] Senior Chemist, Organic Plastics Section, National Bureau of Standards, Washington, D. C.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON HIGH ADDITIVE CONTENT OILS

The Symposium on High Additive Content Oils was held at the First Pacific Area National Meeting of the American Society for Testing Materials in San Francisco, Calif., October 10–14, 1949. The oils considered in these papers are of an entirely new type, containing five to ten times as much additive as found in previous heavy-duty oils.

The following papers were presented:

Introduction—A. H. Batchelder
Does Service Performance Justify Higher Quality Lubricating Oils?—W. B. Bassett
High Detergency Motor Oils—A. B. Boehm and C. O. Tongberg
Five Years' Experience with Special Heavy-Duty Oils—J. A. Edgar and R. E. Jeffrey, Jr.
Possible Future Military Engine Oils for Ground Equipment—N. L. Klein

The papers and discussion are issued as A.S.T.M. Special Technical Publication No. 102, entitled, "Symposium on High Additive Content Oils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TURBINE OILS

The Symposium on Turbine Oils was held at the First Pacific Area National Meeting of the American Society for Testing Materials in San Francisco, Calif., October 10–14, 1949. The four papers on different phases of turbine lubrication were designed to review the progress made in the field and to consider future problems.

The papers presented are as follows:

- Introduction—F. C. Linn
- Lubrication Problems and Requirements of Gas Turbine Equipment—F. C. Linn
- Operating Experience and Problems Relative to Lubricating Systems of Steam Turbine Sets for Utilities—V. F. Estcourt
- Specific Problems Pertaining to Lubrication of Industrial Turbines—A. S. Morrow
- Lubrication of Marine Turbine Propulsion Equipment—Frederick S. Jones

These papers together with discussions are issued as A.S.T.M. Special Technical Publication No. 105, entitled, "Symposium on Turbine Oils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON THE IDENTIFICATION AND CLASSIFICATION OF SOILS

This Symposium on the Identification and Classification of Soils, designed to present a description of the most widely used procedures for identifying and classifying soils for engineering purposes, was held at the Fifty-third Annual Meeting of the American Society for Testing Materials in Atlantic City, N. J., June 29, 1950, under the sponsorship of Committee D-18 on Soils for Engineering Purposes.

The following papers were presented:

Introduction—George A. McAlpin
Identification and Classification of Soils—An Appraisal and Statement of Principles—Donald M. Burmister
A Standard Classification of Soils as Proposed by the Bureau of Reclamation—E. A. Abdun-Nur
Discussion of the Classification of Highway Subgrade Materials Initiated by the Highway Research Board—W. F. Abercrombie
Soil Classification for Highway Purposes—Edward A. Willis
Soil Series Names as a Basis for Interpretive Soil Classifications for Engineering Purposes—Earl J. Felt

These papers together with discussions are issued as A.S.T.M. Special Technical Publication No. 113, entitled, "Symposium on the Identification and Classification of Soils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON TRIAXIAL TESTING OF SOILS AND BITUMINOUS MIXTURES

The Symposium on Triaxial Testing of Soils and Bituminous Mixtures, as published, will combine papers presented at the Symposium on Testing Soils Under Triaxial Loading, held at the Fifty-third Annual Meeting of the Society in Atlantic City, N. J., June 26–30, 1950, under the sponsorship of Committee D-18 on Soils for Engineering Purposes and those presented at a session on Bituminous Paving Mixtures, held at the First Pacific Area National Meeting in San Francisco, Calif., October 10–14, 1949.

Combining these two groups of papers into one publication has seemed logical and desirable. The relationship is very close, not only in the fact that the same type of apparatus is used and physical characteristics measured with both soils and bituminous materials but also in that the two fields of materials are closely related and the same research and testing personnel in most cases are interested in the findings of all of the papers. In fact, the Annual Meeting symposium papers include coverage of bituminous mixtures as well as of soils.

Introduction—W. G. Holtz
History and Theory of Triaxial Testing and the Preparation of Realistic Test Specimens—V. A. Endersby
Applications of the Triaxial Test to Bituminous Mixtures—California Research Corp. Method—V. R. Smith
Some Concepts Concerning Triaxial Compression Testing of Asphaltic Paving Mixtures and Subgrade Materials—H. E. Davis and F. N. Hveem
Application of Triaxial Testing to the Design of Bituminous Pavements—N. W. McLeod
Triaxial Design Correlated with Flexible Pavement Performance in Kansas—H. E. Worley
Triaxial Testing, Adapted to Soils, Flexible Base, and Foundations—R. J. Hank
A Triaxial Apparatus for Testing Large Soil Specimens—E. B. Hall
Shear Testing at the Waterways Experimental Stations—S. J. Johnson
A Triaxial Shear Investigation on a Partially Saturated Soil—D. W. Taylor
Shear Characteristics of Remolded Earth Materials—A. A. Wagner
The Importance of Natural Controlling Conditions Upon Triaxial Compression Test Conditions—D. M. Burmister
Application of Triaxial Compression Test Results to Highway Soil Problems—E. S. Barber and C. L. Sawyer

The combined papers of both meetings are issued as A.S.T.M. Technical Publication No. 106, entitled, "Symposium on Triaxial Testing of Soils and Bituminous Mixture."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON APPLICATION OF STATISTICS

The technique of applying statistical methods to quality control received widespread attention during World War II and since that time has enjoyed rapid growth. This Symposium, presented at the First Pacific Area National Meeting of the American Society for Testing Materials in San Francisco, Calif., October 10–14, 1949, was designed to present some of its practical problems as well as to introduce the subject to those unfamiliar with it.

The following papers were presented:

Introduction—C. M. Wakeman
The Economic Relationship Between Design and Acceptance Specifications—Eugene L. Grant
Precision and Accuracy of Test Methods—Grant Wernimont
Use of Statistics to Determine Precision of Test Methods—W. J. Youden and J. M. Cameron
Modern Quality Control—Introduction by Simon Collier to Motion Picture

These papers together with discussions are issued as A.S.T.M. Special Technical Publication No. 103, entitled, "Symposium on Application of Statistics."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON DYNAMIC STRESS DETERMINATIONS

Recent years have witnessed considerable development of new techniques and types of equipment for making and interpreting dynamic measurements. This Symposium on Dynamic Stress Determination, held at the First Pacific Area National Meeting in San Francisco, Calif., October 10–14, 1949, was designed to assist those who are interested either in initiating such tests or in the practical application of the results of such tests.

The following papers were presented:

Introduction—H. D. Eberhart
Analysis and Interpretation of Dynamic Records—H. C. Roberts
Survey of Pickups for Dynamic Physical Measurements—G. E. White
Selection of Recording Equipment for Dynamic Testing—K. D. Swartzel
Some Examples of Dynamic Testing in the Field of Materials and Structures—Douglas McHenry and H. E. Hosticka

These papers together with discussions are issued as A.S.T.M. Special Technical Publication No. 104, entitled, "Symposium on Dynamic Stress Determinations."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON THE RÔLE OF NON-DESTRUCTIVE TESTING IN THE ECONOMICS OF PRODUCTION

This Symposium, held at the Fourth and Seventh Sessions of the Fifty-third Annual Meeting of the American Society for Testing Materials in Atlantic City, N. J., June 27, 1950, under the sponsorship of Committee E-7 on Non-Destructive Testing, presented papers and discussions on the value of the various recognized methods of non-destructive testing in the economic aspects of production and in promoting the quality of materials.

The following papers were presented:

Introduction—H. H. Lester
A Basic Guide for Management's Choice of Non-Destructive Tests—Robert C. McMaster and Samuel A. Wenk
Discontinuities in Cast and Wrought Products That Can Be Revealed by Non-Destructive Tests—Kent R. Van Horn
The Rôle of Non-Destructive Testing in the Economics of Castings—John W. Juppenlatz
The Economics and Practical Application of Cobalt 60 in the Radiographic Inspection of Steel Weldments—W. L. Schwinn
The Economics of Wrought Steel Inspection—C. D. Moriarty
Management's Responsibility for Insistence on Non-Destructive Testing in the Development of New Engineering Products and Processes—L. W. Ball

These papers and discussions are issued as A.S.T.M. Special Technical Publication No. 112, entitled, "Symposium on the Rôle of Non-Destructive Testing in the Economics of Production."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ULTRASONIC TESTING

A session on Ultrasonic Testing was sponsored by Committee E-7 on Non-Destructive Testing at the Fifty-second Annual Meeting of the American Society for Testing Materials held in Atlantic City, N. J. on June 28, 1949. The theme of the papers is that ultrasonic testing is being used extensively, is practical, and is an aid to manufacturing processes as well as being an inspection tool.

The following papers are included:

Introduction—H. C. Amtsberg
Ultrasonic Wave Propagation in Materials: A Guide to Theoretical Results—Louis Gold
Inspection, Processing, and Manufacturing Control of Metals by Ultrasonic Methods—Carlton H. Hastings and Seymour W. Carter
Basic Principles of Ultrasonic Testing—John C. Smack
Ultrasonics in the Heavy Forging Industry—James C. Hartley
The Application of Ultrasonics to the Fabrication of Aluminum—J. V. Carroll
Ultrasonic Testing in Railroad Work—E. D. Hall
Ultrasonics in the Electrical Industry—D. M. Kelman
Ultrasonic Testing of Bronze Forgings and Ingots—A. Piltch
Pulse Techniques Applied to Dynamic Testing—J. R. Leslie
The Measurement of Dynamic Modulus in Adhesive Joints at Ultrasonic Frequencies—A. G. H. Dietz, P. J. Closmann, G. M. Kavanagh, and J. N. Rossen

The papers together with discussions are issued as A.S.T.M. Special Technical Publication No. 101, entitled, "Symposium on Ultrasonic Testing."